Supporting Information for:

Oxorhenium(V) Complexes with Pyrazole Based Aryloxide Ligands and Application in Olefin Metathesis

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Molecular Structures for $[ReOBr_2(L1)(PPh_3)]$ (**1**), $[ReOBr_2(L4)(PPh_3)]$ (**4**), $[ReOBr_2(L6)(PPh_3)]$ (**6**), $[ReOBr_2(L7)(PPh_3)]$ (**7**), $[ReOCl(L1)_2]$ (**8**), $[ReO(L1)_2]^+(CF_3SO_3)^-$ (**8a**), $[ReOCl(L4)_2]$ (**10**) and $[ReOCl(L6)_2]$ (**11**).

Molecular Structures of the Compounds. Structures of compounds **1**, **4**, **6**, **7**, **8**, **8a**, **10**, and **11** were determined by X–ray diffraction analyses. Selected bond lengths and angles for **1, 4, 6** and **7** are given in **Table S1,** for **8** and **8a** in

Table S2 and **10** and **11** in **Table S3**. Crystallographic acquisition parameters for **1** and **4** are given in **Table S4**, for **6** and **7** in **Table S5**, for **8** and **8a** in **Table S6** and for **10** and **11** in **Table S7**. All complexes show a six coordinate rhenium atom with distorted octahedral geometries and *trans* O–Re=O and *cis* halide–Re=O bonds (except cationic compound **8a**). The *trans* coordination of the ligand oxygen towards the oxygen atom attached to the rhenium is well documented in the literature.[1-3] All complexes gave bond lengths for Re=O (1.6912(19) Å **1**, 1.6900(18) Å **4**; 1.6957(13) Å **6**; 1.6955(17) Å **7**; 1.687(2) Å **8**; 1.690(2) Å **8a**; 1.6994(19) Å **10**; 1.684(5) Å **11**) and Re-Br (2.5009(2) and 2.5642(3) Å **1**, 2.5246(3) and 2.5668(3) Å **4**; 2.4990(2) and 2.4796(4) Å **6**; and 2.5826(3) and 2.4958(3) Å **7**) or Re–Cl (2.3725(9) **8**; 2.37378(7) Å **10**, 2.3689(12) **11**) within the expected range for other $[ReOX_2L(PPh_3)]$ or $[ReOXL_2]$ (X= Cl or Br) structures.[4-9]

The equatorial plane of the monosubstituted complexes **1**-**7** consist of the two bromine atoms, the phosporous atom and the nitrogen atom of the pyrazole ligand. In complex **1**, the bromine atoms Br(1) and Br(2) are coordinated in *cis* position to each other with bond lengths of Re(1)-Br(1) 2.5642(3) and Re(1)-Br(2) 2.5009(2) Å including an angle Br(1)-Re(1)-Br(2) of 86.842(8)°. The nitrogen atom N(1) is in *trans* position to the bromine atom Br(2) with an angle of N(1)-Re(1)-Br(2) 169.32(6)°, the phosphorus atom P(1) is in *trans* position to the bromine atom Br(1) with an angle of P(1)-Re(1)-Br(1) 176.560(16)°. The phenol oxygen O(2) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(2) of 166.41(8)°. The pyrazol ring and the phenolate ring of the attached ligand include a dihedral angle of 15.23°.

In complex **4**, bromine atoms Br(1) and Br(2) are coordinated in *cis* position to each other with bond lengths of Re(1)-Br(1) 2.5668(3) and Re(1)-Br(2) 2.5246(3) Å including an angle Br(1)-Re(1)-Br(2) of 89.265(8)°. The nitrogen atom N(1) is in *trans* position to the bromine atom Br(2) with an angle of N(1)-Re(1)-Br(2) 162.94(6)°, the phosphorus atom P(1)

is in *trans* position to the bromine atom Br(1) with an angle of P(1)-Re(1)-Br(1) 174.313(16)°. The naphthol oxygen O(2) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(2) of 173.06(8)°. The pyrazol ring and the naphtholate ring of the attached ligand include a dihedral angle of 9.91°.

In complex **6**, bromine atoms Br(1) and Br(2) are coordinated in *cis* position to each other with bond lengths of Re(1)-Br(1) 2.4796(4) and Re(1)-Br(2) 2.4990(2) Å including an angle Br(1)-Re(1)-Br(2) of 89.013(7)°. The nitrogen atom N(1) is in *trans* position to the bromine atom Br(2) with an angle of N(1)-Re(1)-Br(2) 169.69(4)°, the phosphorus atom P(1) is in *trans* position to the bromine atom Br(1) with an angle of P(1)-Re(1)-Br(1) 178.507(12)°. The naphthol oxygen O(2) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(2) of 165.91(6)°. The pyrazol ring and the naphtholate ring of the attached ligand include a dihedral angle of 36.47°.

In complex **7**, bromine atoms Br(1) and Br(2) are coordinated in *cis* position to each other with bond lengths of Re(1)-Br(1) 2.5826(3) and Re(1)-Br(2) 2.4958(3) Å including an angle Br(1)-Re(1)-Br(2) of 89.938(9)°. The nitrogen atom N(1) is in *trans* position to the bromine atom Br(2) with an angle of N(1)-Re(1)-Br(2) 168.49(5)°, the phosphorus atom P(1) is in *trans* position to the bromine atom Br(1) with an angle of P(1)-Re(1)-Br(1) 177.096(15)°. The naphthol oxygen O(2) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(2) of 165.52(7)°. The pyrazol ring and the naphtholate ring of the attached ligand include a dihedral angle of 36.27°.

The equatorial plane of the disubstituted complexes **8**, **8a**, **10**, and **11** consist of the halide atom (nitrogen atom from ACN for **8a**) and the two nitrogen atoms and an oxygen atom from the ligands. In complex **8**, nitrogen atoms N(1) and N(3) of the ligands are coordinated in *cis* position to each other with bond lengths of Re(1)-N(1) 2.146(2) and Re(1)-N(3) 2.128(3) Å including an angle N(1)-Re(1)-N(3) of 95.82(9)°. The nitrogen atom N(1) of

one ligand is in *trans* position to the oxygen atom O(3) of the second ligand with an angle of N(1)-Re(1)-O(3) 164.95(9)°, the nitrogen atom N(3) of one ligand is in *trans* position to the chlorine atom Cl(1) with an angle of N(3)-Re(1)-Cl(1) 172.62(7)°. The phenol oxygen O(2) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(2) of 166.69(10)°. The nitrogen atoms and the oxygen atom of the ligands attached to the metal centre include angles of N(1)-Re(1)-O(2) 81.13(9)° and N(3)-Re(1)-O(3) 88.40(9)°. The pyrazol ring and the phenolate ring of the attached ligands include dihedral angles of 24.81° and 13.75°.

In complex **8a**, nitrogen atoms N(1) and N(3) of the ligands are coordinated in *trans* position to each other with bond lengths of Re(1)-N(1) 2.073(3) and Re(1)-N(3) 2.125(3) Å including an angle N(1)-Re(1)-N(3) of 168.42(10)°. The residual nitrogen N(1S) from the coordinated acetonitril has bond length of Re(1)-N(1S) 2.149(3) Å to N(1) and N(3) of the pyrazole ligands and is in *trans* position to the oxygen atom O(2) of the pyrazole ligand with an angle of N(1S)-Re(1)-O(2) 171.87(10)°, the phenol oxygen O(3) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(3) of 162.75(10)°. The nitrogen atoms and the oxygen atom of the ligands attached to the metal centre include angles of N(1)-Re(1)-O(2) 86.51(10)° and N(3)-Re(1)-O(3) 81.36(10)°. The pyrazol ring and the phenolate ring of the attached ligands include dihedral angles of 20.17° and 20.00°.

In complex **10**, nitrogen atoms N(5) and N(7) of the ligands are coordinated in *cis* position to each other with bond lengths of Re(2)-N(5) 2.115(2) and Re(2)-N(7) 2.119(3) Å including an angle N(5)-Re(2)-N(7) of 97.71(9)°. The nitrogen atom N(7) of one ligand is in *trans* position to the oxygen atom O(5) of the second ligand with an angle of N(7)-Re(2)-O(5) 164.29(9)°, the nitrogen atom N(5) of one ligand is in *trans* position to the chlorine atom Cl(2) with an angle of N(3)-Re(2)-Cl(1) 170.24(7)°. The naphthol oxygen O(6) shows a *trans* coordination towards the Re=O unit with an angle of O(4)-Re(2)-O(6) of 164.75(9)°. The

nitrogen atoms and the oxygen atom of the ligands attached to the metal centre include angles of N(7)-Re(2)-O(6) 80.84(9)° and N(5)-Re(2)-O(5) 87.59(9)°. The pyrazol ring and the naphtholate ring of the attached ligands include dihedral angles of 20.42° and 14.97°.

In complex **11**, nitrogen atoms N(1) and N(3) of the ligands are coordinated in *cis* position to each other with bond lengths of Re(1)-N(1) 2.140(5) and Re(1)-N(3) 2.117(4) Å including an angle N(1)-Re(1)-N(3) of 94.89(17)°. The nitrogen atom N(3) of one ligand is in *trans* position to the oxygen atom O(2) of the second ligand with an angle of N(3)-Re(1)-O(2) 163.84(15)°, the nitrogen atom N(1) of one ligand is in *trans* position to the chlorine atom Cl(1) with an angle of N(1)-Re(1)-Cl(1) 168.97(13)°. The naphthol oxygen O(3) shows a *trans* coordination towards the Re=O unit with an angle of O(1)-Re(1)-O(3) of 164.3(2)°. The nitrogen atoms and the oxygen atom of the ligands attached to the metal centre include angles of N(1)-Re(1)-O(2) 87.10(16)° and N(3)-Re(1)-O(3) 79.56(16)°. The pyrazol ring and the naphtholate ring of the attached ligands include dihedral angles of 36.61° and 38.15°.

Table S1. Selected Bond Distances (Å) and Angles (°) for $[ReOBr_2(L1)PPh_3]$ (**1**), $[ReOBr_2(L4)PPh_3]$ (**4**), $[ReOBr_2(L6)PPh_3]$ (**6**), $[ReOBr_2(L7)PPh_3]$ (**7**).

	1	**4**	**6**	**7**
Re(1)–O(1)	1.6912(19)	1.6900(18)	1.6957(13)	1.6955(17)
Re(1)–O(2)	1.9277(18)	1.9240(17)	1.9354(12)	1.9221(16)
Re(1)–N(1)	2.161(2)	2.160(2)	2.1630(15)	2.1486(19)
Re(1)–P(1)	2.4817(6)	2.4737(6)	2.4796(4)	2.5826(3)
Re(1)–Br(2)	2.5009(2)	2.5246(3)	2.4990(2)	2.4958(3)
Re(1)–Br(1)	2.5642(3)	2.5668(3)	2.4796(4)	2.4648(6)
O(1)–Re(1)–O(2)	166.41(8)	173.06(8)	165.91(6)	165.52(7)
Br(1)–Re(1)–P(1)	176.560(16)	174.313(16)	169.69(4)	168.49(5)
Br(2)–Re(1)–N(1)	169.32(6)	162.94(6)	178.507(12)	177.096(15)
Br(1)–Re(1)–Br(2)	86.842(8)	89.265(9)	89.013(7)	89.938(9)
O(2)–Re(1)–N(1)	80.19(8)	79.12(8)	79.77(5)	78.78(7)
O(1)–Re(1)–P(1)	84.50(7)	81.93(6)	82.38(4)	85.94(6)
P(1)–Re(1)–N(1)	93.55(6)	89.29(5)	92.60(4)	98.04(6)
P(1)–Re(1)–O(2)	86.12(5)	92.09(5)	88.37(4)	90.90(5)

Table S2. Selected Bond Distances (Å) and Angles (°) for $[ReOCl(L1)_2]$ (**8**), $[ReO(L1)_2]^+(CF_3SO_3)^-$ (**8a**)

	8		**8a**
Re(1)–O(1)	1.687(2)	Re(1)–O(1)	1.690(2)
Re(1)–O(2)	1.955(2)	Re(1)–O(3)	1.959(2)
Re(1)–N(3)	2.128(3)	Re(1)–O(2)	1.968(2)
Re(1)–O(3)	1.988(2)	Re(1)–N(1)	2.073(3)
Re(1)–Cl(1)	2.3725(9)	Re(1)–N(1S)	2.149(3)
Re(1)–N(1)	2.146(2)	Re(1)–N(3)	2.125(3)
O(1)–Re(1)–O(2)	166.69(10)	O(1)–Re(1)–O(3)	162.75(10)
N(3)–Re(1)–Cl(1)	172.62(7)	O(2)–Re(1)–N(1S)	171.87(10)
N(1)–Re(1)–O(3)	164.95(9)	N(3)–Re(1)–N(1)	168.42(10)
O(1)–Re(1)–Cl(1)	100.16(8)	O(1)–Re(1)–N(1S)	84.67(11)
N(3)–Re(1)–O(3)	88.40(9)	N(1)–Re(1)–O(2)	86.51(10)
N(1)–Re(1)–O(2)	81.13(9)	N(3)–Re(1)–O(3)	81.36(10)
Cl(1)–Re(1)–O(2)	91.58(6)	N(1S)–Re(1)–N(1)	98.59(10)
N(1)–Re(1)–N(3)	95.82(9)	N(1S)–Re(1)–N(3)	86.69(10)

Table S3. Selected Bond Distances (Å) and Angles (°) for [ReOCl(L4)$_2$] (**10**) and [ReOCl(L6)$_2$] (**11**)

	10		**11**
Re(2)–O(4)	1.6994(19)	Re(1)–O(1)	1.684(5)
Re(2)–O(6)	1.9911(19)	Re(1)–O(3)	2.003(4)
Re(2)–N(5)	2.115(2)	Re(1)–N(3)	2.117(4)
Re(2)–O(5)	1.969(2)	Re(1)–Cl(1)	2.3689(12)
Re(2)–Cl(2)	2.3738(7)	Re(1)–O(2)	1.977(4)
Re(2)–N(7)	2.119(3)	Re(1)–N(1)	2.140(5)
O(4)–Re(2)–O(6)	164.57(9)	O(1)–Re(1)–O(3)	164.3(2)
N(5)–Re(2)–Cl(2)	170.24(7)	N(3)–Re(1)–O(2)	163.84(15)
N(7)–Re(2)–O(5)	164.29(9)	N(1)–Re(1)–Cl(1)	168.97(13)
O(4)–Re(2)–Cl(2)	101.62(7)	O(1)–Re(1)–Cl(1)	101.53(17)
N(5)–Re(2)–O(5)	87.59(9)	N(1)–Re(1)–O(2)	87.10(16)
N(7)–Re(2)–O(6)	80.84(9)	N(3)–Re(1)–O(3)	79.56(16)
Cl(2)–Re(2)–O(5)	85.00(6)	Cl(1)–Re(1)–O(2)	83.78(11)
N(5)–Re(2)–N(7)	97.71(9)	N(1)–Re(1)–N(3)	94.89(17)

Table S4. Crystallographic Acquisition Parameters for **1** and **4**

	1	**4**
formula	$C_{31}H_{29}Br_2Cl_2N_3O_2PRe$	$C_{36}H_{34}BrN_2O_3PRe$
fw [g/mol]	923.46	919.64
color/habit	green/column	green / parallelepiped
crystal size [mm^3]	0.4 x 0.07 x 0.04	045 x 0.15 x 0.06
crystal system	triclinic	monoclinic
space group	P-1	P2(1)/c
a [Å]	11.1389(5)	10.9168(5)
b [Å]	12.0044(5)	17.8373(9)
c [Å]	13.7105(5)	17.4947(9)
α [deg]	79.480(2)	90.00
β [deg]	87.674(2)	101.340(2)
γ [deg]	63.668(2)	90.00
V, [$Å^3$]	1613.8(1)	3340.2(3)
Z	2	4
T [K]	100(2)	100(2)
D_{calcd} [g cm^{-3}]	1.901	1.829
μ [mm^{-1}]	6.490	6.117
F(000)	892	1792
θ range [deg]	2.04 to 30.06	2.28 to 32.04
limiting indices	$-15 \le h \le 15$	$-16 \le h \le 16$
	$-16 \le k \le 16$	$-26 \le k \le 26$
	$-19 \le l \le 19$	$-26 \le l \le 26$
no. reflns collected	53457	135986
no. independent, R_{int}	9232, 0.0327	11559, 0.0524
no. obs. $I>2\sigma(I)$	8472	9647
data/restraints/params	9232 / 0 / 354	11559 / 0 / 407
R1/wR2 ($I>2\sigma(I)$)[a]	0.0270 / 0.0235	0.0402 / 0.0250
R1/wR2 (all data)[a]	0.0607 / 0.0594	0.0540 / 0.0471
GOF (on F^2)[a]	1.042	1.107
largest diff peak/hole [e $Å^{-3}$]	1.657 and -1.689	1.809 and -1.789

[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $GOF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$.

Table S5. Crystallographic Acquisition Parameters for **6** and **7**

	6	**7**
formula	$C_{32}H_{26}Br_2N_2O_2PRe$	$C_{35}H_{34}Br_2N_2O_3PRe$
fw [g/mol]	847.54	907.63
color/habit	green / block	green / column
crystal size [mm^3]	0.2 x 0.15 x 0.10	0.36 x 0.10 x 0.08
crystal system	monoclinic	monoclinic
space group	P21/c	P2(1)/c
a [Å]	15.0730(7)	16.4921(7)
b [Å]	12.8745(6)	10.8415(5)
c [Å]	15.3274(7)	18.6374(9)
α [deg]	90.00	90.00
β [deg]	109.515(2)	99.714(2)
γ [deg]	90.00	90.00
V, [$Å^3$]	2803.5(2)	3284.6(3)
Z	4	4
T [K]	100(2)	100(2)
D_{calcd} [g cm^{-3}]	2.008	1.835
μ [mm^{-1}]	7.276	6.219
F(000)	1632	1768
θ range [deg]	2.12 to 37.23	2.18 to 33.62
limiting indices	$-25 \le h \le 24$	$-25 \le h \le 19$
	$-20 \le k \le 21$	$-16 \le k \le 15$
	$-25 \le l \le 26$	$-28 \le l \le 29$
no. reflns collected	184234	81813
no. independent, R_{int}	14516, 0.0440	12967, 0.0483
no. obs. I>2σ(I)	11421	10394
data/restraints/params	14516 / 0 / 362	12967 / 0 / 443
R1/wR2 (I>2σ(I))[a]	0.0408 / 0.0241	0.0448 / 0.0270
R1/wR2 (all data)[a]	0.0489 / 0.0438	0.0590 / 0.0520
GOF (on F^2)[a]	1.054	1.054
largest diff peak/hole [e $Å^{-3}$]	1.737 and -1.054	2.849 and -1.729

[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $GOF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$.

Table S6. Crystallographic Acquisition Parameters for **8** and **8a**

	8	**8a**
formula	$C_{22}H_{21}ClN_5O_3Re$	$C_{23}H_{21}F_3N_5O6ReS$
fw [g/mol]	625.09	738.71
color/habit	green / needle	green / needle
crystal size [mm^3]		0.40 x 0.05 x 0.05
crystal system	monoclinic	monoclinic
space group	P2(1)/n	P2(1)/n
a [Å]	7.8444(16)	12.3618(5)
b [Å]	10.133(2)	13.9869(5)
c [Å]	27.976(6)	14.1942(5)
α [deg]	90.00	90.00
β [deg]	90.36(3)	94.6470(10)
γ [deg]	90.00	90.00
V, [$Å^3$]	2223.7(8)	2446.16(16)
Z	4	4
T [K]	133(2)	100(2)
D_{calcd} [g cm^{-3}]	1.867	2.006
μ [mm^{-1}]	5.619	1440
F(000)	1216	5.128
θ range [deg]	1.46 to 24.79	2.05 to 30.02
limiting indices	$-8 \le h \le 9$	$-17 \le h \le 17$
	$-11 \le k \le 11$	$-19 \le k \le 19$
	$-32 \le l \le 32$	$-19 \le l \le 19$
no. reflns collected	12164	50786
no. independent, R_{int}	3796, 0.0295	7126, 0.0367
no. obs. I>2σ(I)	3332	6244
data/restraints/params	3796 / 0 / 292	7126 /0 / 355
R1/wR2 (I>2σ(I))[a]	0.0227 / 0.0174	0.0320 / 0.0242
R1/wR2 (all data)[a]	0.0396 / 0.0385	0.0580 / 0.0545
GOF (on F^2)[a]	1.009	1.160
largest diff peak/hole [e $Å^{-3}$]	0.441 and -0.527	2.727 and -0.901

[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $GOF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$.

Table S7. Crystallographic Acquisition Parameters for **10** and **11**

	10	**11**
formula	$C_{57}H_{46}Cl_4N_8O_6Re_2$	$C_{31}H_{28}Cl_4N_4O_{3.50}Re$
fw [g/mol]	1453.22	840.57
color/habit	green / tablet	green / platelet
crystal size [mm^3]	0.06 x 0.08 x 0.20	0.5 x 0.4 x 0.03
crystal system	triclinic	monoclinic
space group	P-1	C2/c
a [Å]	10.3040(3)	34.108(4)
b [Å]	16.1853(6)	12.7947(16)
c [Å]	16.8848(6)	15.1945(19)
α [deg]	72.512(2)	90.00
β [deg]	85.9850(10)	107.172(5)
γ [deg]	75.6200(10)	90.00
V, [$Å^3$]	2601.60(15)	6335.4(14)
Z	2	8
T [K]	100(2)	100(2)
D_{calcd} [g cm^{-3}]	1.855	1.763
μ [mm^{-1}]	4.916	4.215
F(000)	1420	3304
θ range [deg]	2.04 to 30.76	2.09 to 31.08
limiting indices	$-14 \le h \le 13$	$-48 \le h \le 49$
	$-23 \le k \le 23$	$-18 \le k \le 17$
	$-24 \le l \le 24$	$-21 \le l \le 22$
no. reflns collected	52437	96602
no. independent, R_{int}	15974, 0.0377	9892, 0.0466
no. obs. I>2σ(I)	12631	8101
data/restraints/params	15974 / 2 / 726	9892 / 4 / 396
R1/wR2 (I>2σ(I))[a]	0.0425 / 0.0257	0.0697 / 0.0552
R1/wR2 (all data)[a]	0.0559 / 0.0495	0.1963 / 0.1759
GOF (on F^2)[a]	1.026	1.075
largest diff peak/hole [e $Å^{-3}$]	1.364 and -1.100	3.905 and -3.383

[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $GOF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$.

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