Supporting Information for

Synthesis, Characterization, and Structure Determination of Bis-oxazolidine Complexes of Rhenium.

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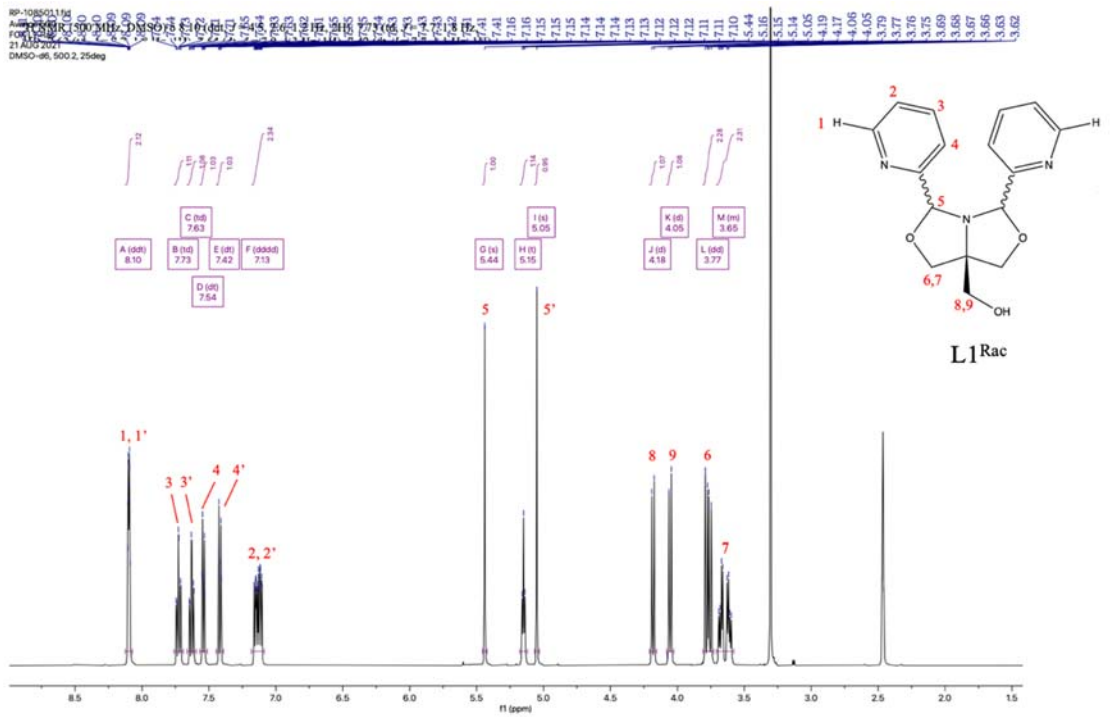


Figure S-1. ^{1}H NMR (500 MHz, DMSO-d_6) spectrum of **L1^{rac}**.

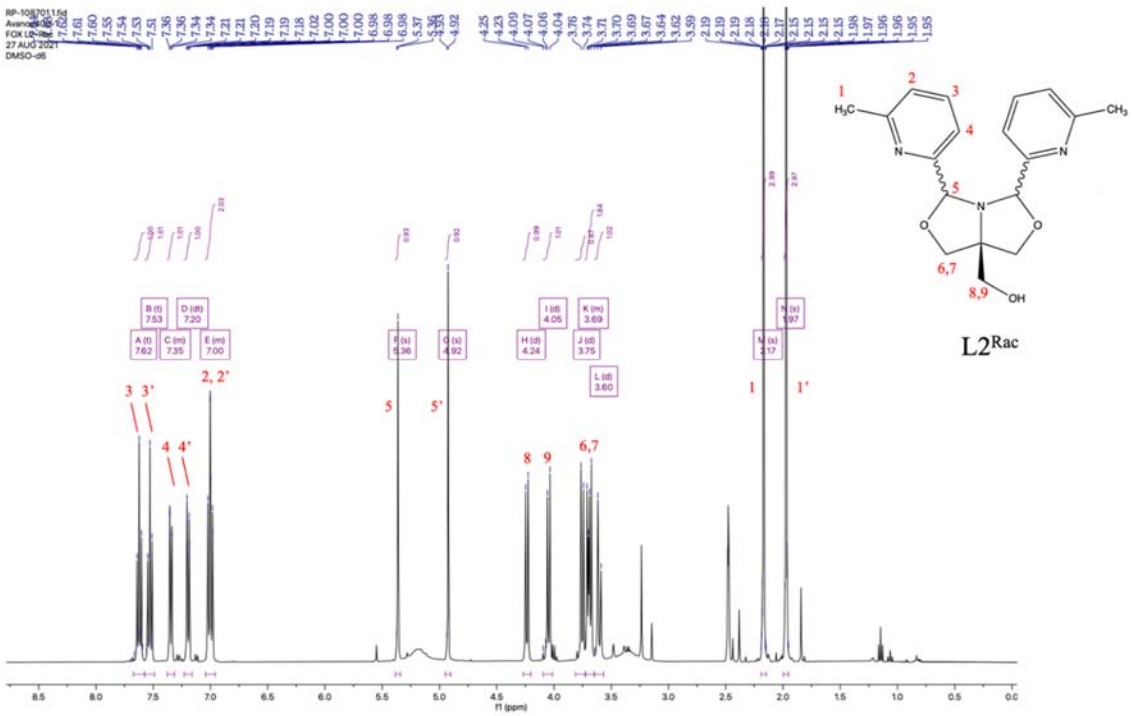


Figure S-2. ^{1}H NMR (400 MHz, DMSO-d_6) spectrum of **L2^{rac}**.

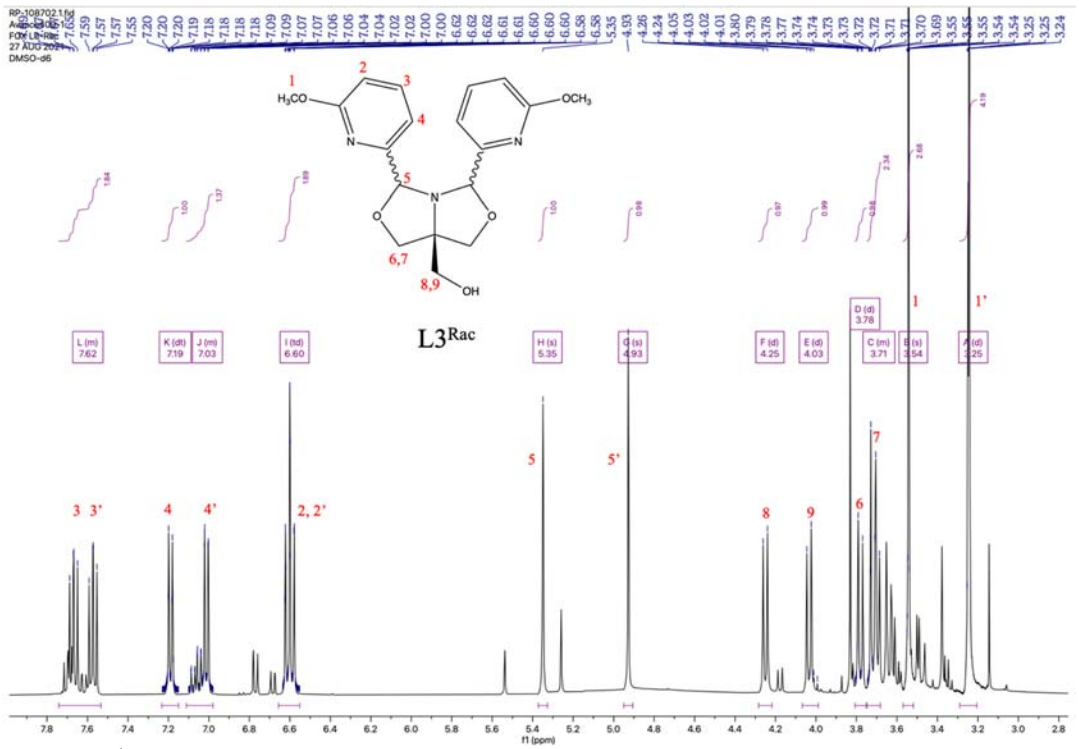


Figure S-3. ^{1}H NMR (400 MHz, DMSO-d_6) spectrum of **L3^{rac}**.

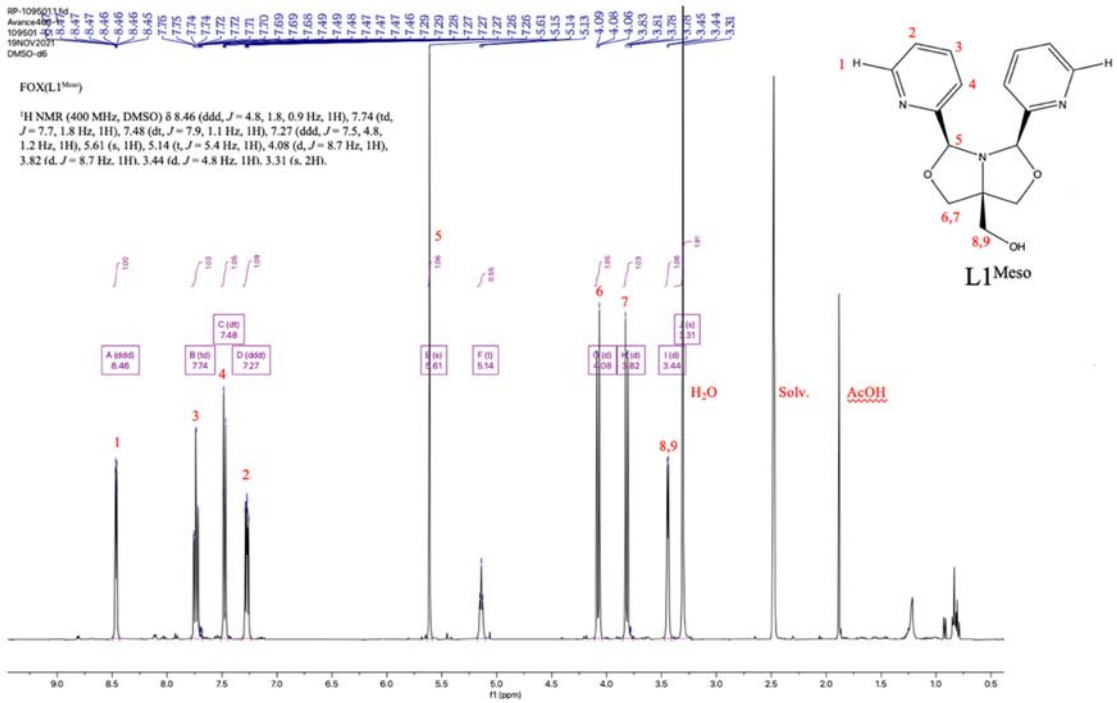


Figure S-4. ^{1}H NMR (400 MHz, DMSO-d_6) spectrum of **L1^{meso}**.

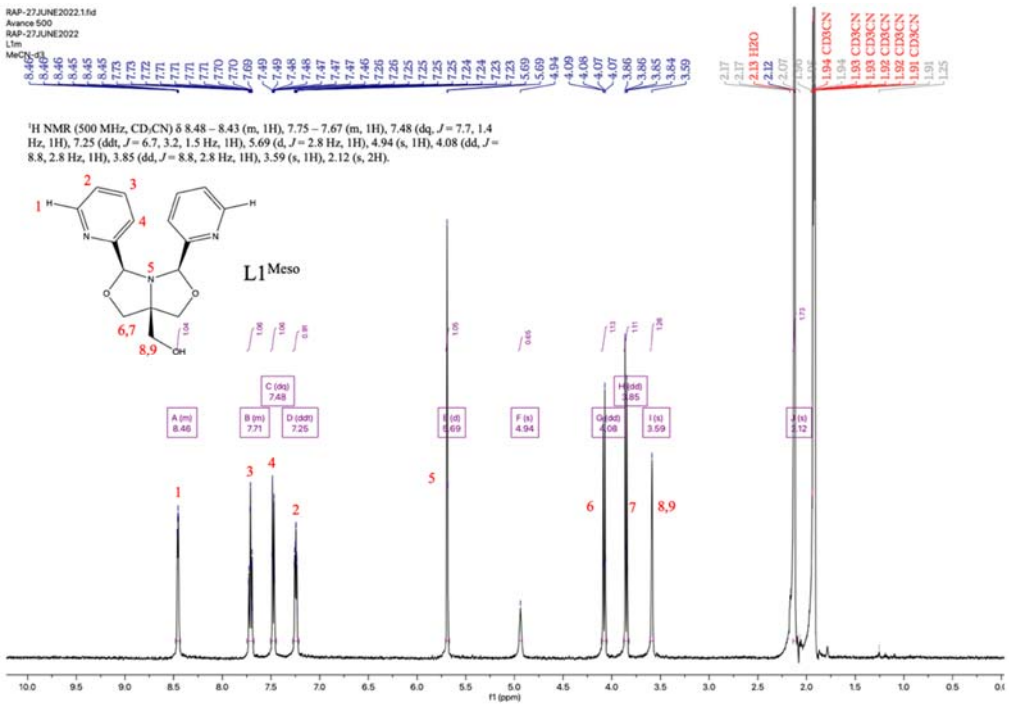


Figure S-5. ^{1}H NMR (500 MHz, MeCN-d_3) spectrum of **L1^{meso}**.

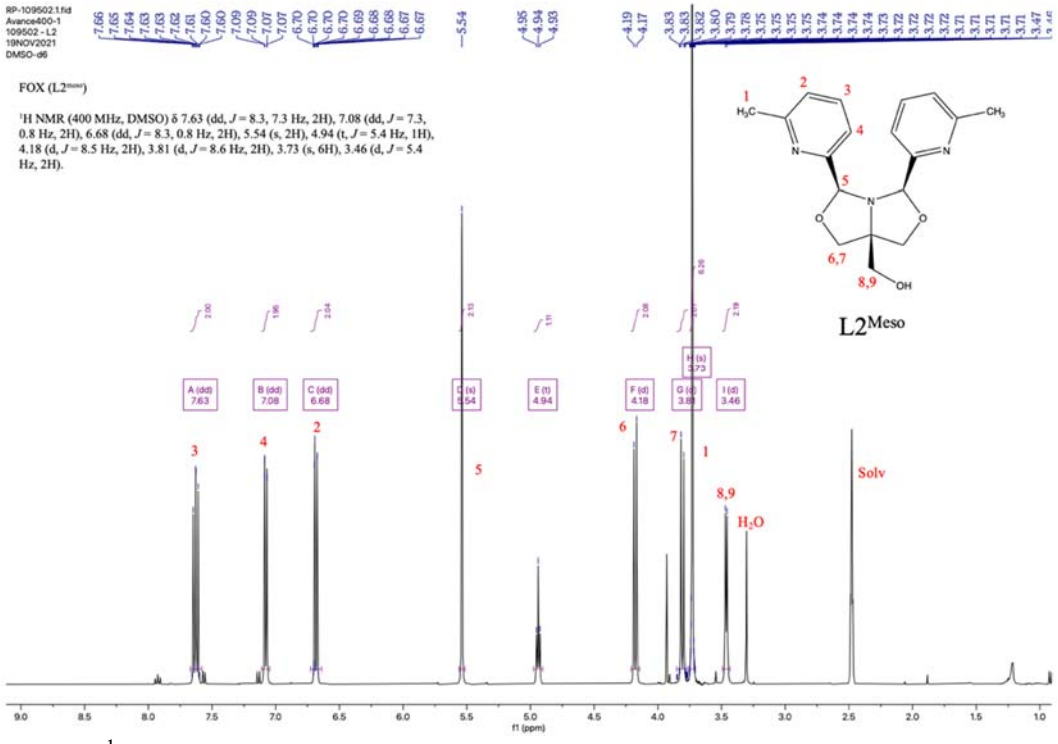


Figure S-6. ^{1}H NMR (500 MHz, DMSO-d_6) spectrum of **L2^{meso}**.

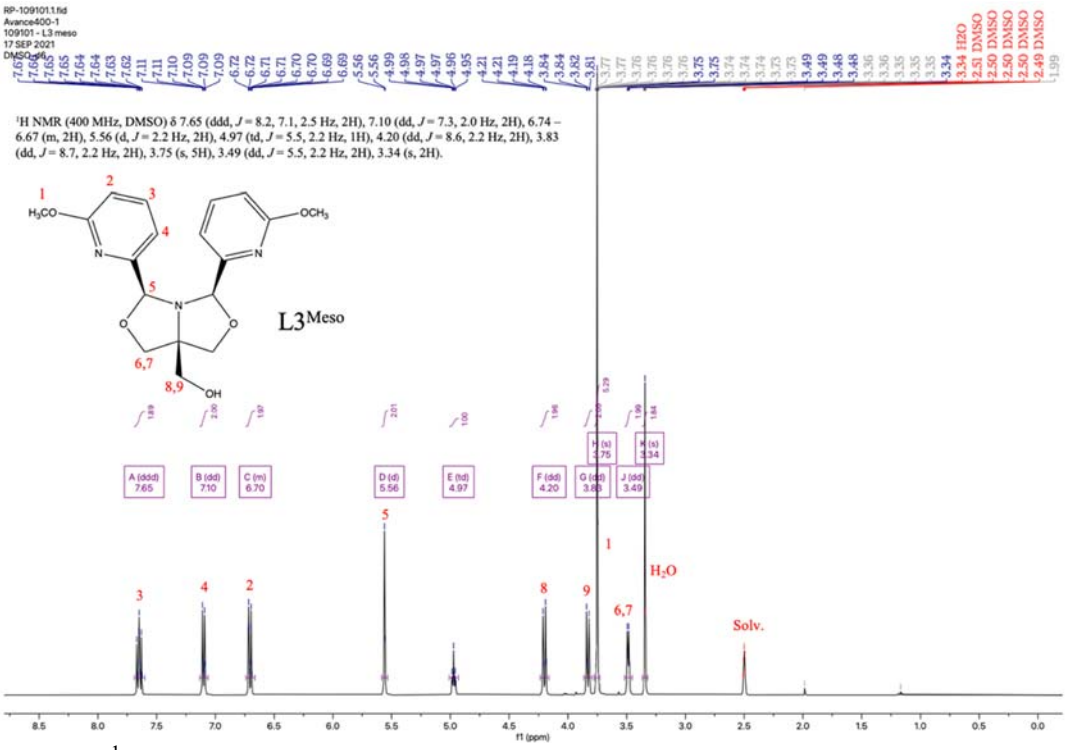


Figure S-7. ^{1}H NMR (400 MHz, DMSO-d_6) spectrum of **L3^{meso}**.

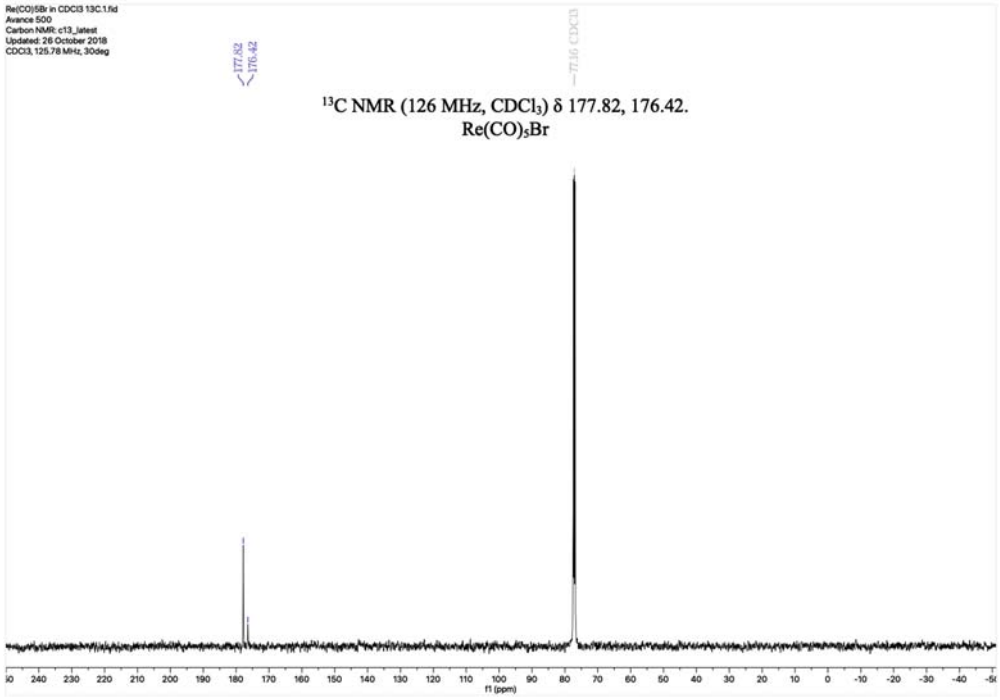


Figure S-8. ^{13}C NMR (126 MHz, $CDCl_3$) spectrum of $Re(CO)_5Br$.

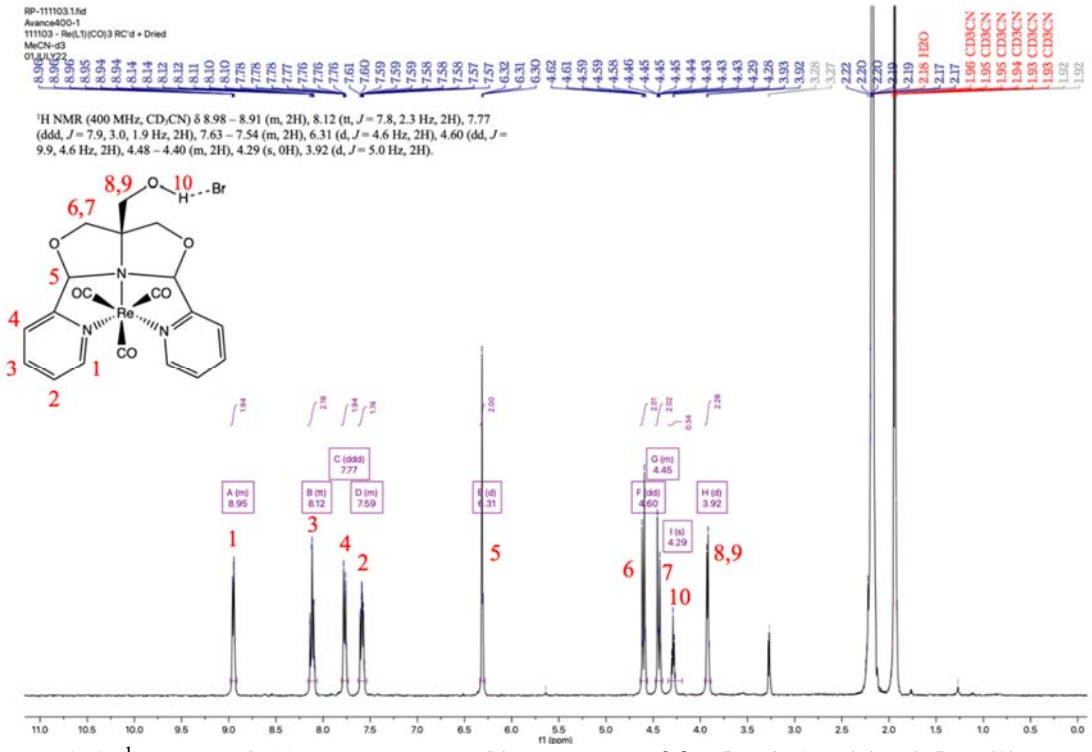


Figure S-9. ^{1}H NMR (500 MHz, MeCN-d_3) spectrum of *fac*-[Re(L1^{meso})(CO)$_3$]Br (**8**).

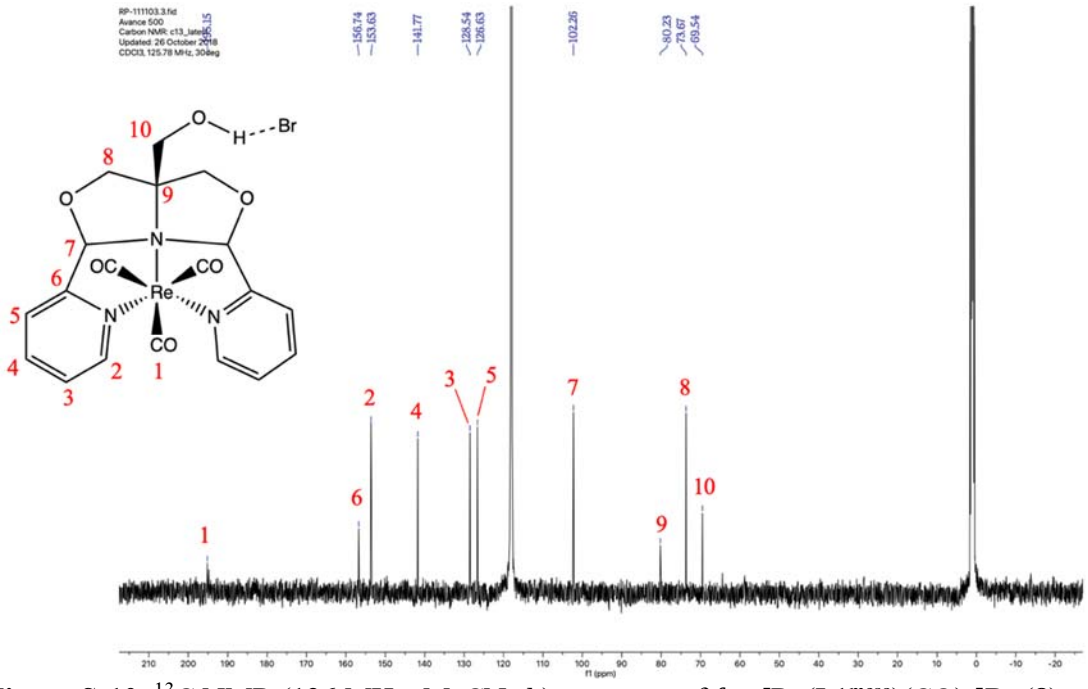


Figure S-10. ^{13}C NMR (126 MHz, MeCN-d_3) spectrum of *fac*-[Re(L1^{meso})(CO)$_3$]Br (**8**).

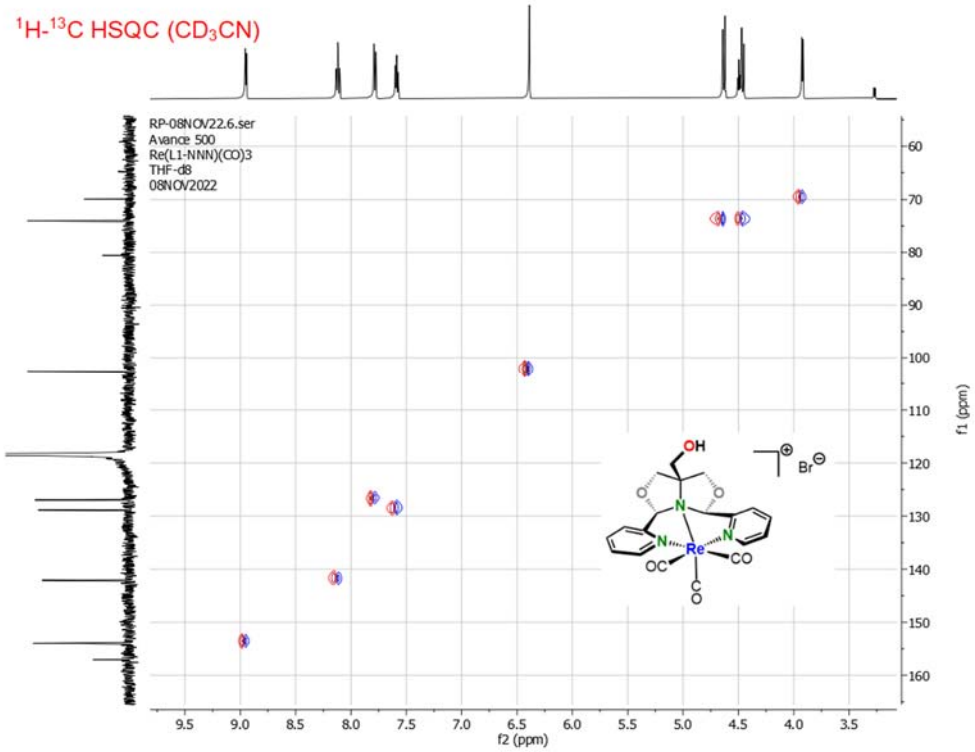


Figure S-11. ^{1}H-^{13}C HSQC (500 MHz, THF-d_8) spectrum of *fac*-[Re(L1^{meso})(CO)$_3$]Br (**8**).

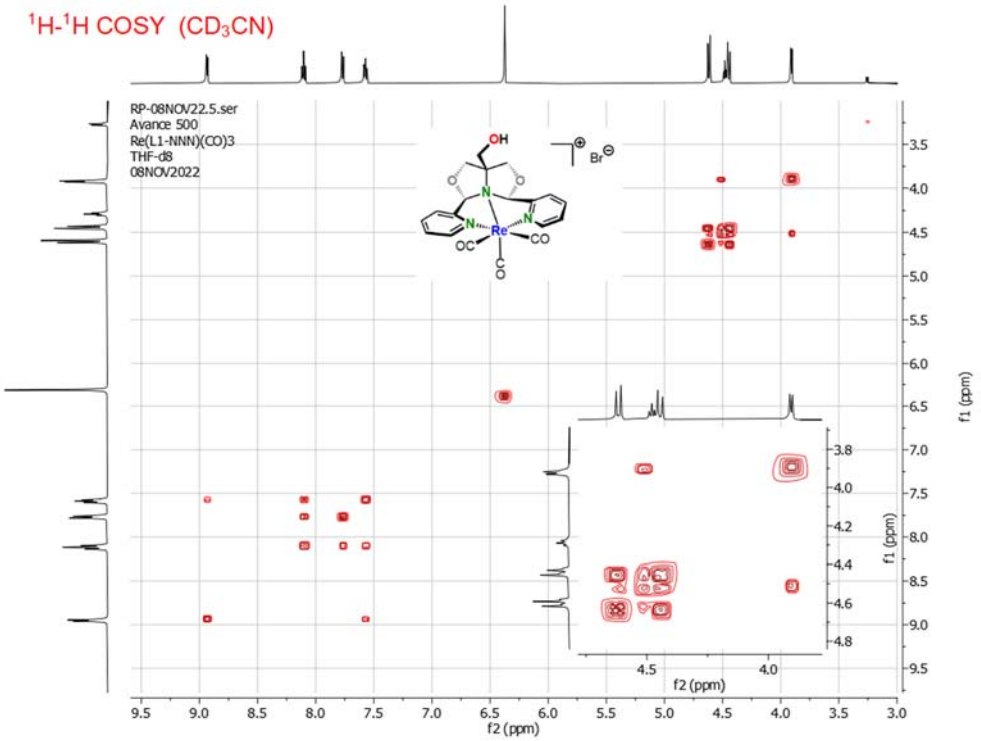


Figure S-12. COSY (500 MHz, THF-d_8) spectrum of *fac*-[Re(L1^{meso})(CO)$_3$]Br (**8**).

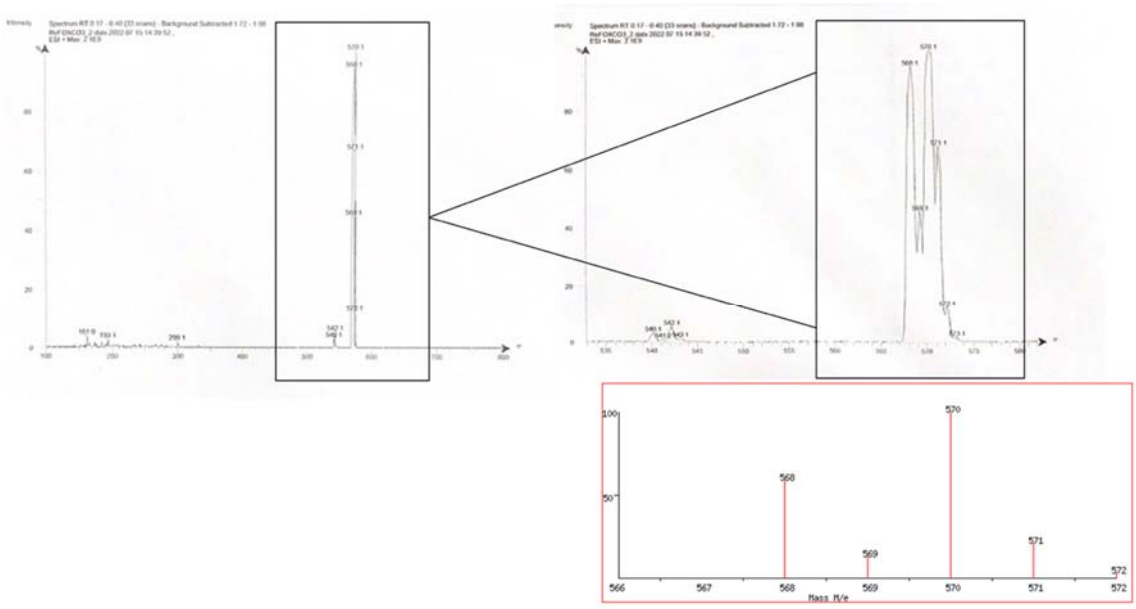


Figure S-13. ESI-MS spectrum of *fac*-[Re(L1^{meso})(CO)$_3$]Br (**8**) (positive ion mode). A simulated spectrum is shown in red. Exact mass (M^+) = 569.56. Observed: 568.1 (M^+ - H^+), 569.1 (M^+), 570.1(M^+ + 1), 568.1 (M^+ + 2).

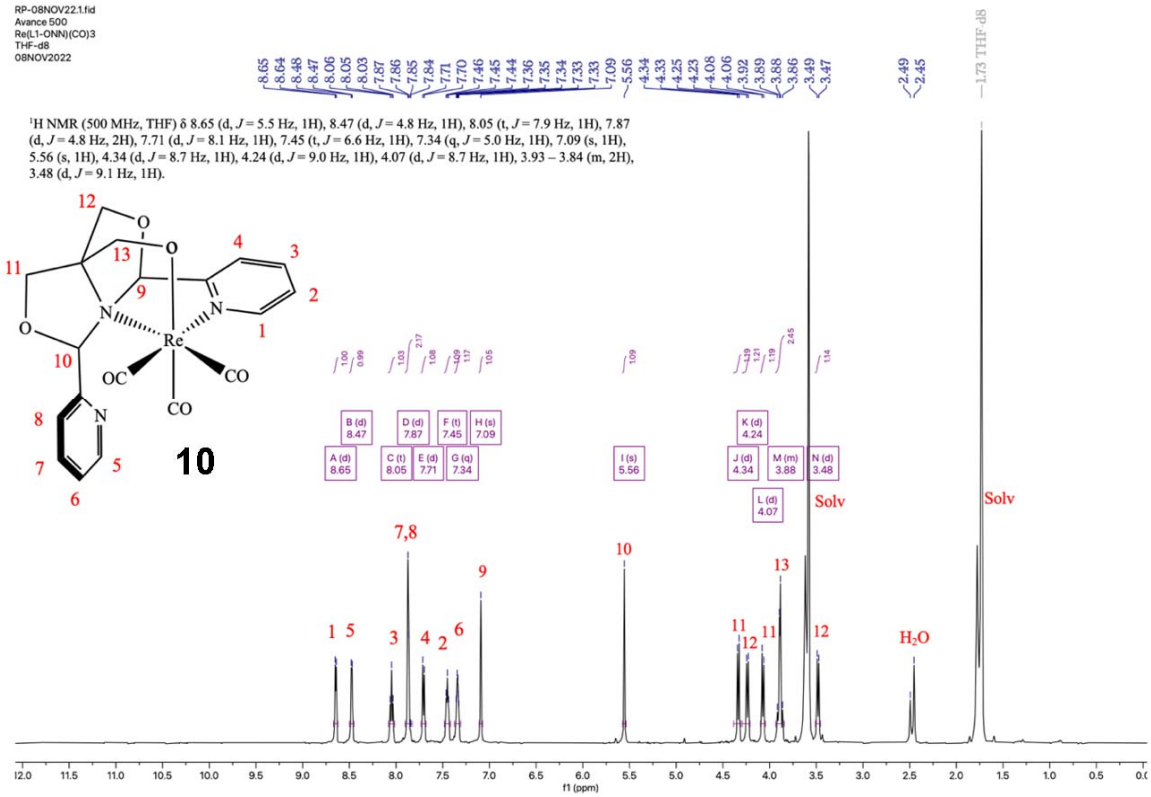


Figure S-14. ^{1}H NMR (500 MHz, THF-d_8) spectrum of *fac*-Re(L1$^{meso\text{-}H}$)(CO)$_3$ (**10**).

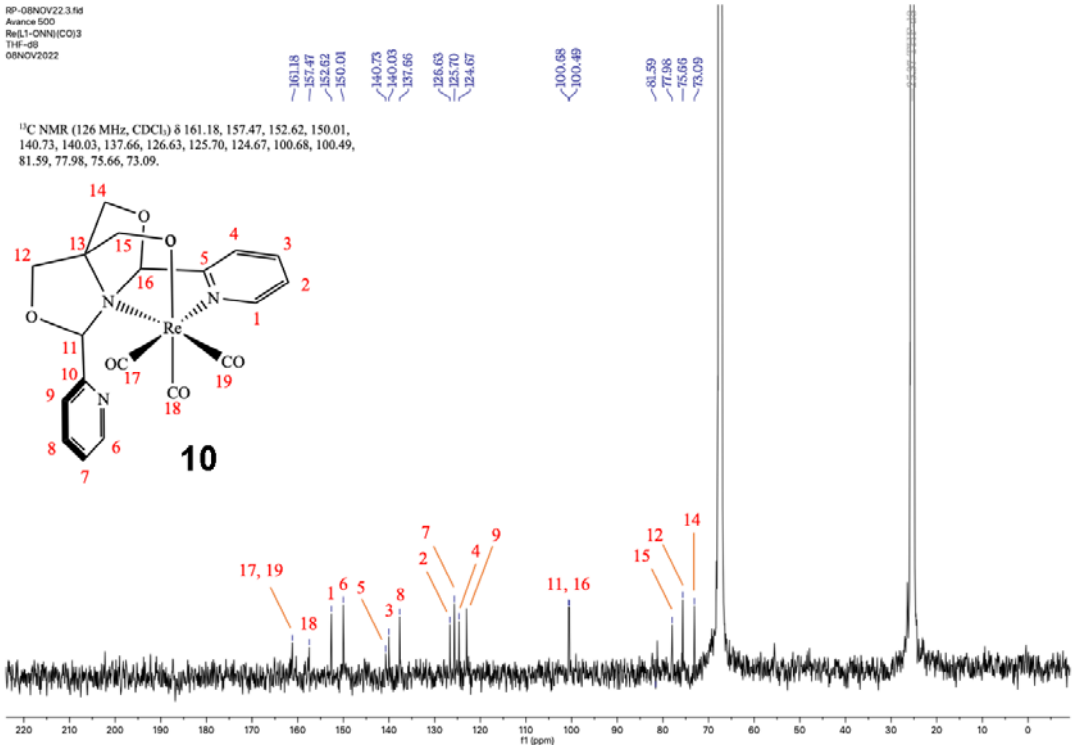


Figure S-15. ^{13}C NMR (126 MHz, THF-d_8) spectrum of *fac*-Re(L1$^{meso\text{-}H}$)(CO)$_3$ (**10**).

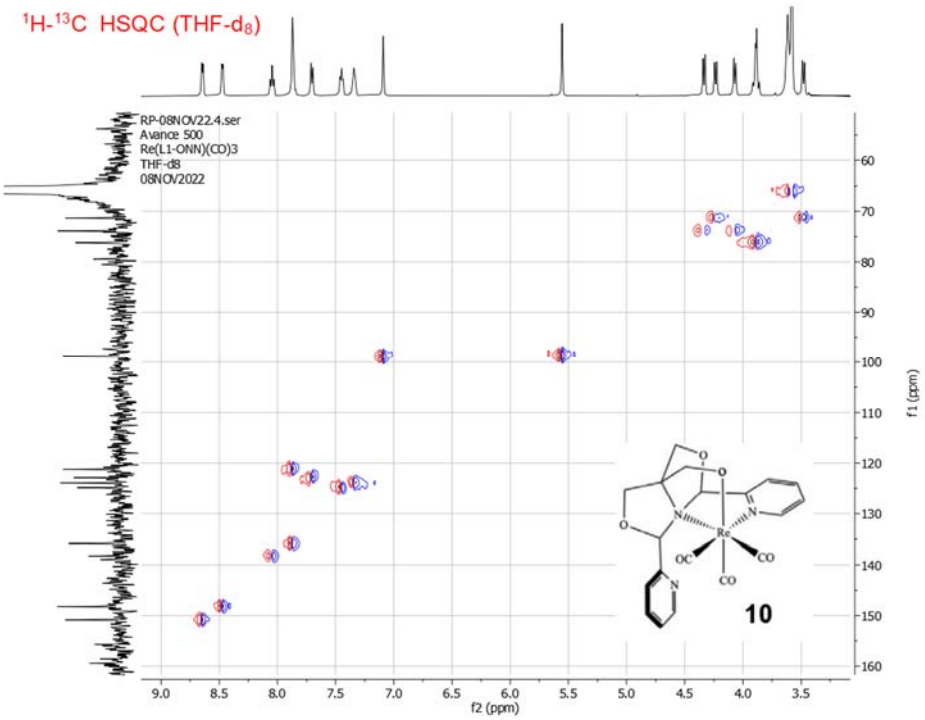


Figure S-16. ^{1}H-^{13}C HSQC (500 MHz, THF-d_8) spectrum of *fac*-Re(L1$^{meso\text{-}H}$)(CO)$_3$ (**10**).

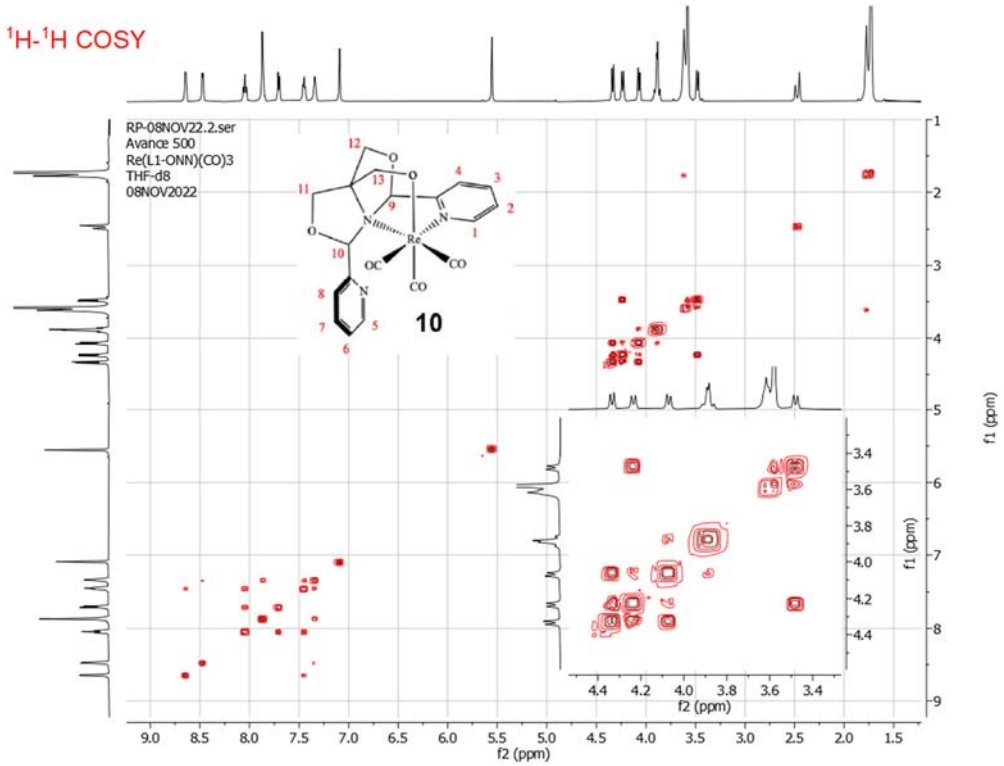


Figure S-17. COSY (500 MHz, THF-d_8) spectrum of *fac*-Re(L1$^{meso\text{-}H}$)(CO)$_3$ (**10**).

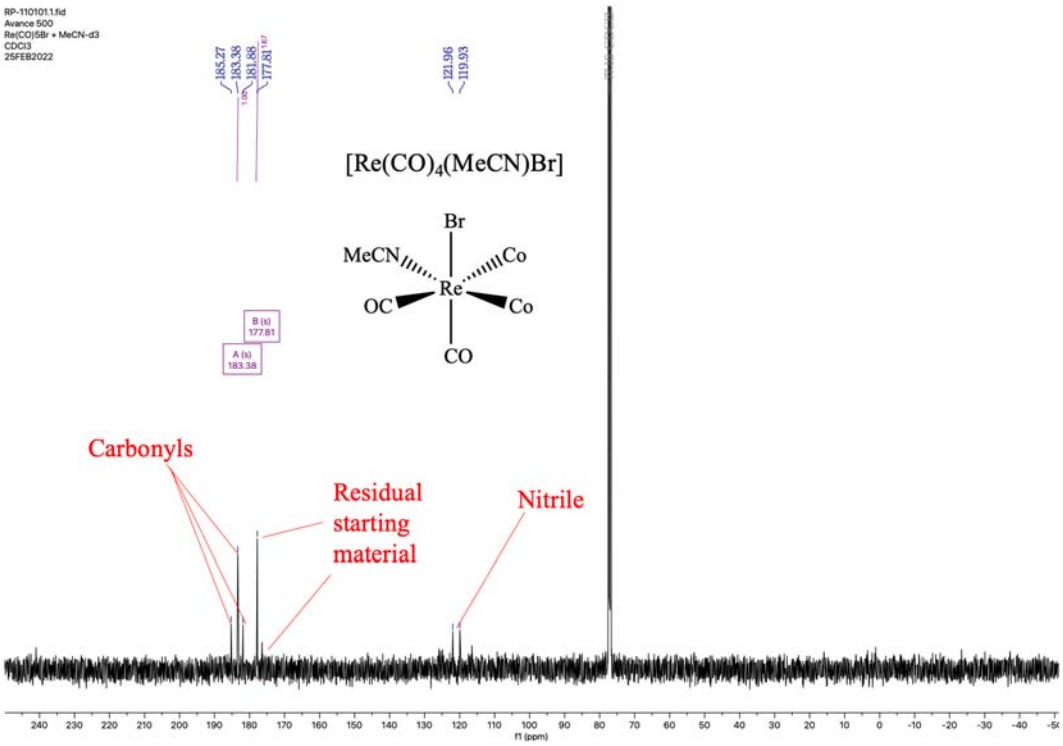


Figure S-19. ^{13}C NMR (126 MHz, $CDCl_3$) spectrum of **[**$Re(CO)_4Br(MeCN)$].

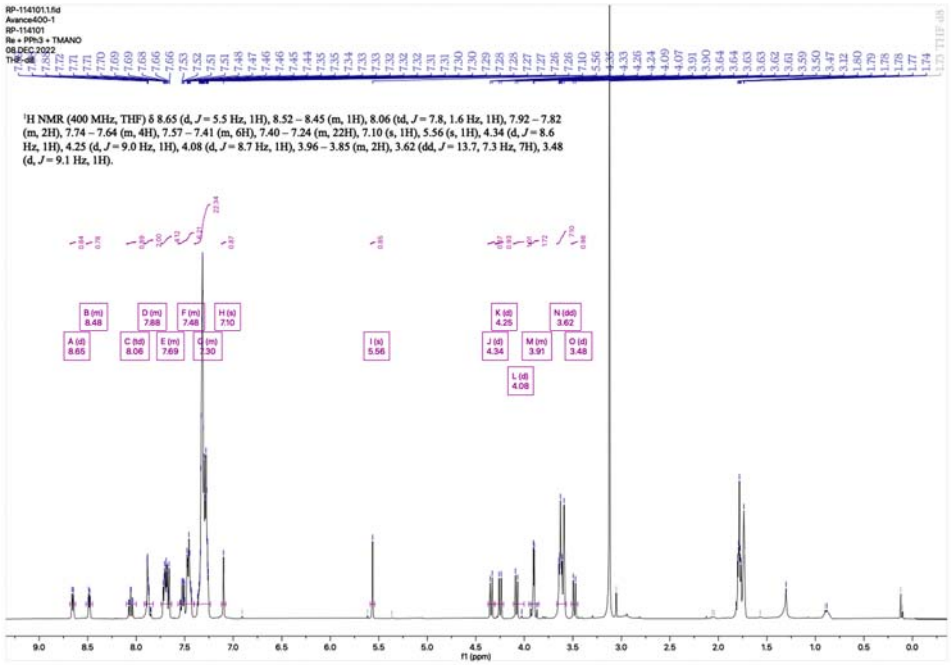
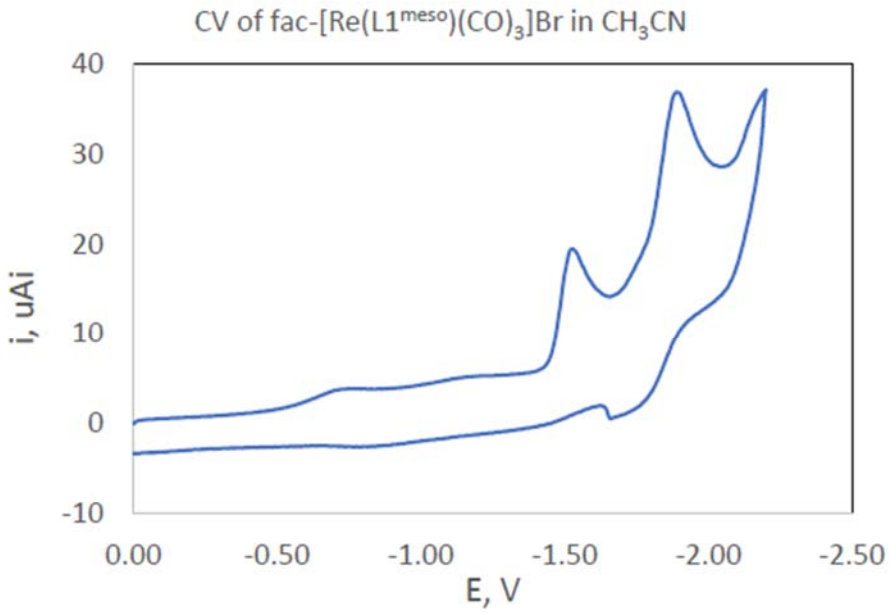


Figure S-20. ^{1}H NMR (400 MHz, THF-d_8) spectrum of product mixture of **10** and PPh_3. No reaction was observed. The spectrum is consistent with **10** and free PPh_3.



Experiment Type : Cyclic Voltammetry (CV)
Title : CV Run for BASi-Epsilon
Data File Name : 218906
Date & Time of the run : 6/19/2023 1:09:10 PM

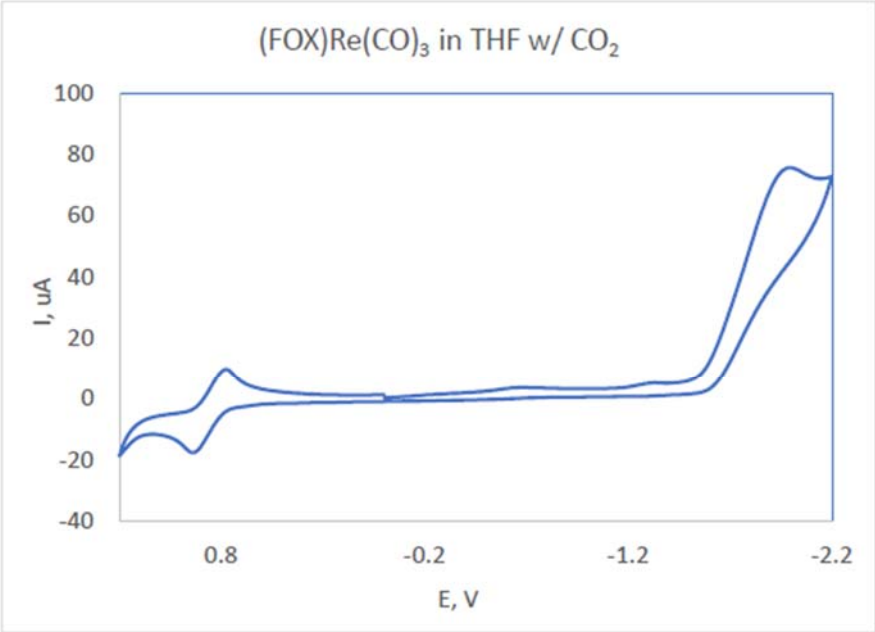
Display Convention : POLAROGRAPHIC
Number of data points : 4400
# of points to skip : 0
Initial Potential : 0 (mV)
Switching Potential 1 : -2200 (mV)
Switching Potential 2 : 0 (mV)
Final Potential : 0 (mV)
Number of segments : 2
Scan rate : 200 (mV/s)
Current Full Scale : 100 uA
Filter : 10 Hz
Quiet Time : 2 (Sec)
Sample Interval : 1 mV

Analyst : wdj
Solution :

Analyte : Re-8
Analyte Conc. : 1mM
Solvent : CH3CN
Supporting Electrolyte : TBAH
S.E. Conc. : 0.1M
pH :
Temperature :
Electrodes :

W.E. material : glassy carbon
W. E. area :
W. E. geometry : round
W. E. radius : 1.5mm
W. E. conditioning :
Reference electrode : Ag wire
Auxiliary electrode : Pt wire
Notes :
CH3CN dried over sieves in air
polish electrode -
100uA

Figures S20. Cyclic voltammogram of **8.**



Epc= -1.874 Ipc= 4.92E-05 49.19
E(Fc) = 0.858 (correct values by -0.858 V to set Fc = 0)

Experiment Type : Cyclic Voltammetry (CV)
Title : CV Run for BASi-Epsilon
Data File Name : 218936
Date & Time of the run : 6/20/2023 10:58:32 AM

Display Convention : POLAROGRAPHIC
Number of data points : 7000
# of points to skip : 0
Initial Potential : 0 (mV)
Switching Potential 1 : -2200 (mV)
Switching Potential 2 : 1300 (mV)
Final Potential : 0 (mV)
Number of segments : 3
Scan rate : 100 (mV/s)
Current Full Scale : 100 uA
Filter : 10 Hz
Quiet Time : 2 (Sec)
Sample Interval : 1 mV

Analyst : wdj
Solution :

Analyte : Re-B
Analyte Conc. : 1.2mM
Solvent : THF
Supporting Electrolyte : TBAH
S.E. Conc. : 0.1M
pH :
Temperature :
Electrodes :

W.E. material : glassy carbon
W. E. area :
W. E. geometry : round
W. E. radius : 1.5mm
W. E. conditioning :
Reference electrode : Ag wire
Auxiliary electrode : Pt wire
Notes :
THF dried over sieves in air
polish electrode -
100uA
Ar
Re-B
w/ CO2
w/Fc
100mV/s

Figures S21. Cyclic voltammogram of **10** with CO_2 and ferrocene.

SHIMADZU LabSolutions **Analysis Report**

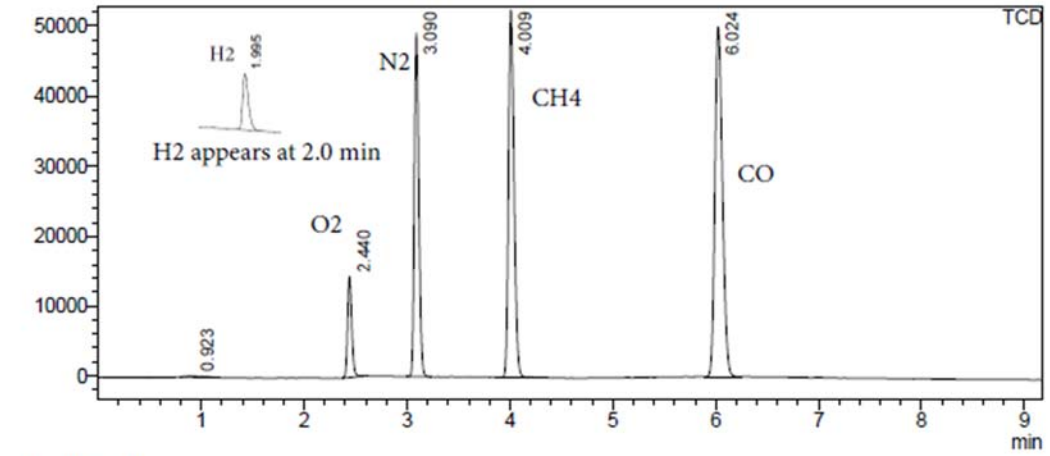
<Sample Information>

Sample Name	: wdj-219313
Sample ID	: wdj-219313
Data Filename	: 219313-TCD.gcd
Method Filename	: tcd2.gcm
Batch Filename	:
Vial #	: 1
Injection Volume	: 100 uL
Date Acquired	: 8/1/2023 4:01:42 PM
Date Processed	: 8/1/2023 4:10:58 PM

Standard Gas Sample:
20 mL CO + 20 mL CH4 + 500 mL CO2
Inject 100 uL

Sample Type	: Standard
Acquired by	: wdjgroup
Processed by	: wdjgroup

<Chromatogram>



<Peak Table>

TCD

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	0.923	2091	150	0.000			
2	2.440	40731	14342	0.000			
3	3.090	149673	48728	0.000			
4	4.009	198481	52270	0.000			methane
5	6.024	254420	49891	0.000			CO
Total		645397	165381				

Response Factor Calculation:

RF = (moles CH4)/(moles CO)(area CH4)/(area CO)
= 1 x (198481)/(254420)
= 0.780

repeated in triplicate - all gave RF = 0.780

Figure S22. GC chromatograms for electrolysis of **10** with CO_2 – standard.

SHIMADZU LabSolutions **Analysis Report**

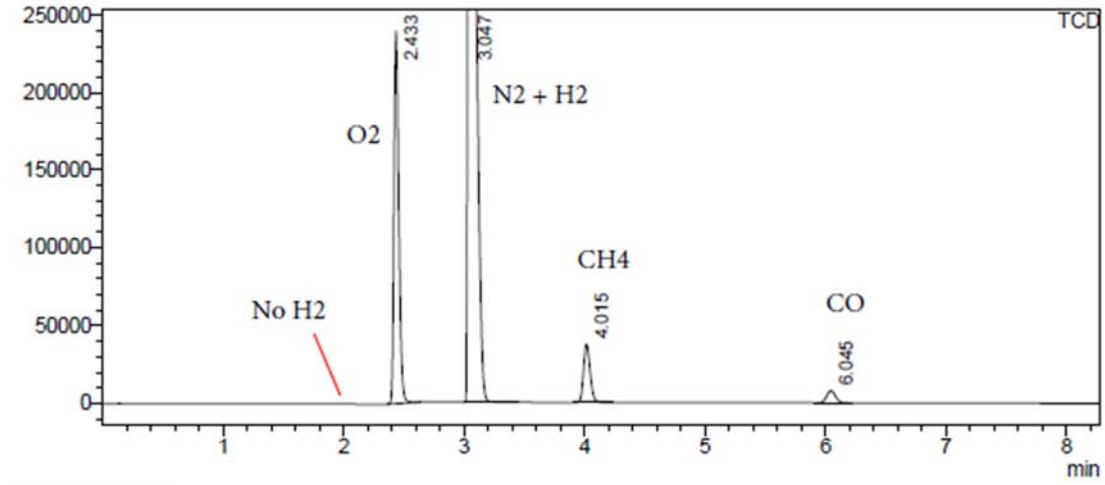
<Sample Information>

Sample Name	: wdj-219310
Sample ID	: wdj-219310
Data Filename	: 219310-TCD.gcd
Method Filename	: tcd2.gcm
Batch Filename	:
Vial #	: 1
Injection Volume	: 100 uL
Date Acquired	: 8/1/2023 3:28:26 PM
Date Processed	: 8/1/2023 3:36:46 PM

Sample of headspace - 200 uL methane added
100 uL injection

Sample Type	: CPE sample
Acquired by	: wdjgroup
Processed by	: wdjgroup

<Chromatogram>



<Peak Table>

TCD

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	2.433	659888	238801	0.000			
2	3.047	2694462	662808	0.000			
3	4.015	141448	37378	0.000		S	
4	6.045	39833	7861	0.000			
Total		3535631	946847				

moles methane added = PV/RT = (1 atm)(0.000200 L)/(0.08205)/(300 K) = 8.13 E-6 moles
moles CO = (RF)(moles CH4)(area CO)/(area CH4)
= (0.780)(8.13E-6)(39833)/(141448) = 1.79 E-6 moles CO

moles Re = 3.5 mg/641 = 5.5 E-6 mol
yield = (1.79E-6)/(5.5E-6) = 32%
3.59 Coulombs passed
=> Faradaic efficiency = (1.79E-6)/(3.59/96500)(2 mole e-/mole CO)=9.6%

Figure S23. GC chromatograms for electrolysis of **10** with CO_2 – sample.

REFERENCE NUMBER: jonrap04

CRYSTAL STRUCTURE REPORT

$C_{19}\ H_{17}\ Br\ N_3\ O_6\ Re$

or

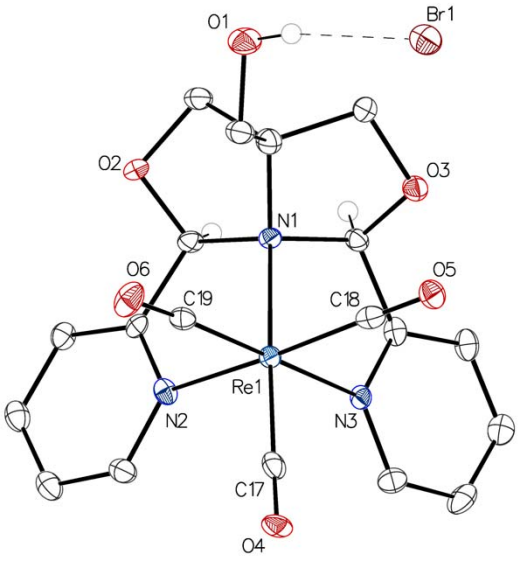
$[(\kappa^3\text{-}L1^{meso})Re(CO)_3][Br]$

(**8**)

Report prepared for:

R. Pohorenec, Prof. W. Jones

November 28, 2021



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.121 x 0.053 x 0.018 mm^3) was placed onto a thin glass optical fiber or a nylon loop and mounted on a Rigaku XtaLAB Synergy-S Dualflex diffractometer equipped with a HyPix-6000HE HPC area detector for data collection at 100.00(10) K. A preliminary set of cell constants and an orientation matrix were calculated from a small sampling of reflections.[1] A short pre-experiment was run, from which an optimal data collection strategy was determined. The full data collection was carried out using a PhotonJet (Cu) X-ray source with frame times of 0.12 and 0.47 seconds and a detector distance of 34.0 mm. Series of frames were collected in 0.50º steps in ω at different 2θ, κ, and ϕ settings. After the intensity data were corrected for absorption, the final cell constants were calculated from the xyz centroids of 37721 strong reflections from the actual data collection after integration.[1] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT[2] and refined using SHELXL.[3] The space group $P2_1/n$ was determined based on systematic absences. Most or all non-hydrogen atoms were assigned from the solution. Full-matrix least squares / difference Fourier cycles were performed which located any remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. The O-H hydrogen atoms were found from the difference Fourier map and refined freely. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0286$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0732$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains two monocationic Re complexes and two Br anions in general positions. The cations and anions are linked via O-H…Br hydrogen bonding (see figures and Table 7).

Structure manipulation and figure generation were performed using Olex2.[4] Unless noted otherwise all structural diagrams containing anisotropic displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B04 Hutchison Hall, Department of Chemistry, University of Rochester. The instrument was purchased with funding from NSF MRI program grant CHE-1725028. .

1 *CrysAlisPro*, version 171.41.120a; Rigaku Corporation: Oxford, UK, 2021.

2 Sheldrick, G. M. *SHELXT*, version 2018/2; *Acta. Crystallogr.* **2015**, *A71*, 3-8.

3 Sheldrick, G. M. *SHELXL*, version 2018/3; *Acta. Crystallogr.* **2015**, *C71*, 3-8.

4 Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *Olex2*, version 1.3-ac4; *J. Appl. Cryst.* **2009**, *42*, 339-341.

Some equations of interest:

$$R_{int} = \Sigma |F_o^2 - < F_o^2 >| / \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$$

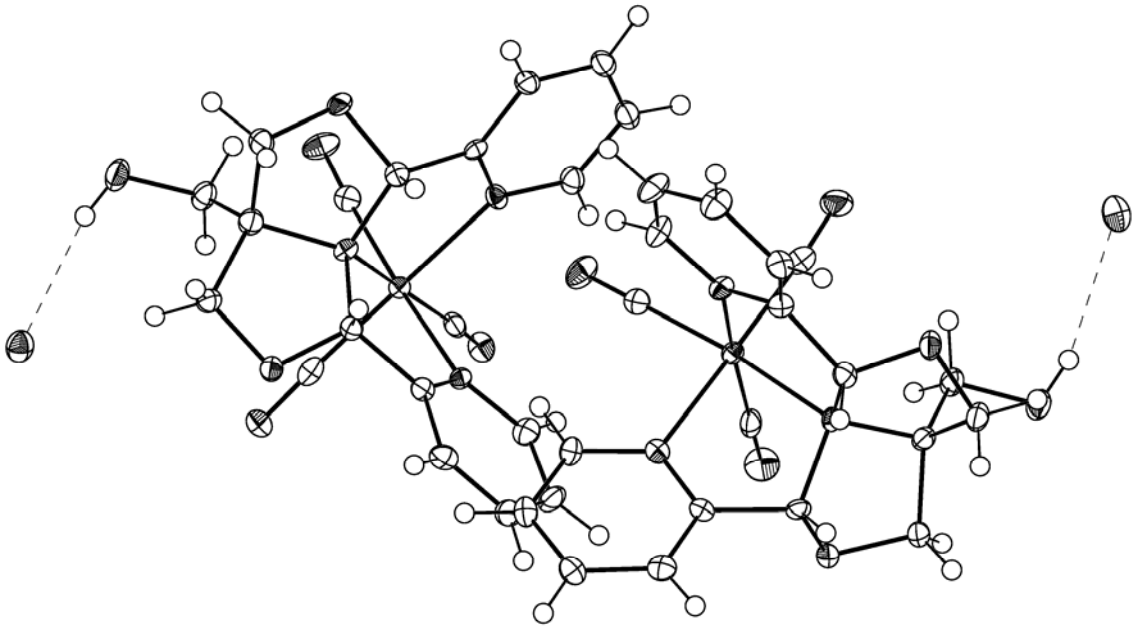
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]]^{1/2}$$

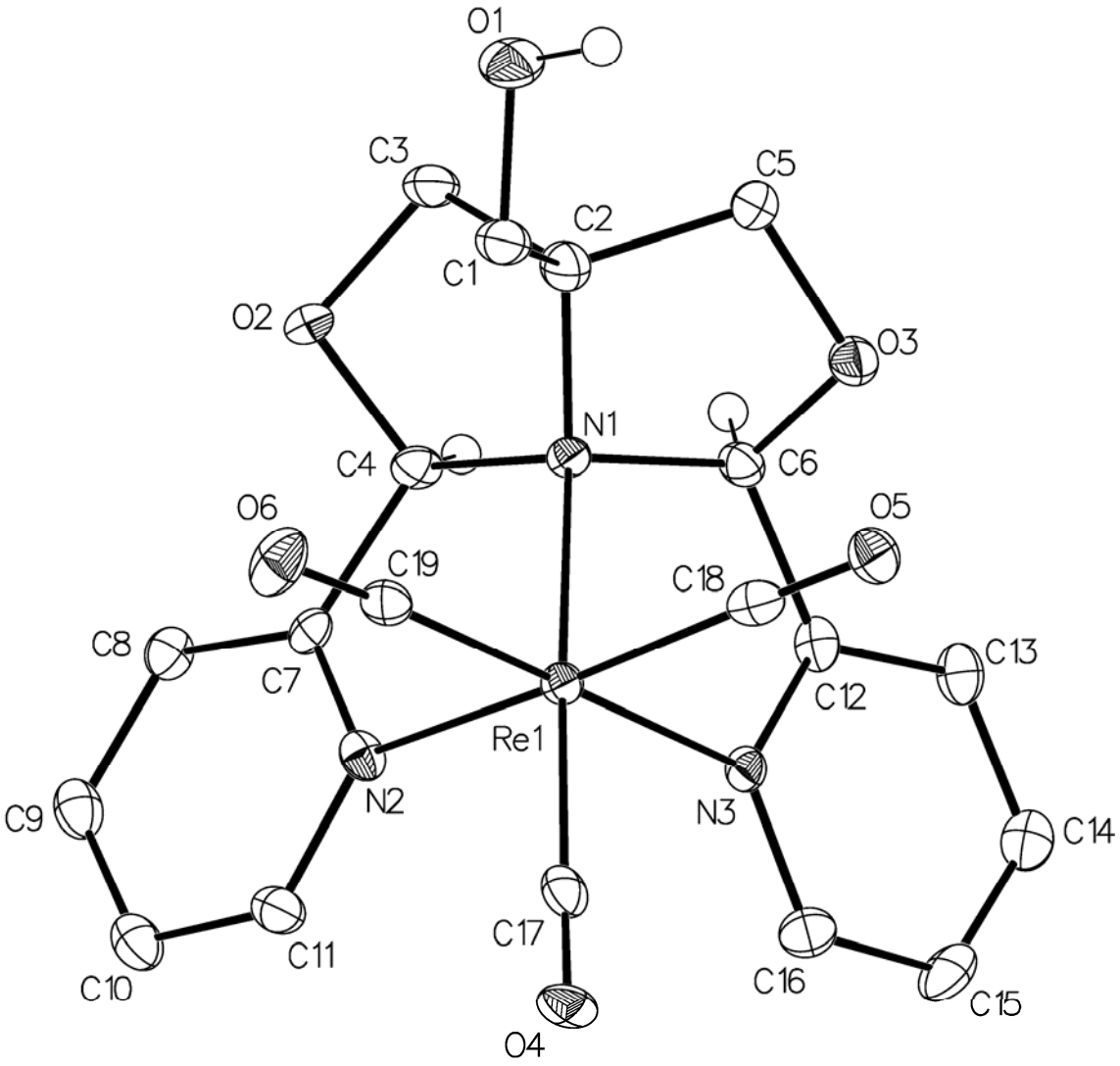
where $w = 1 / [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

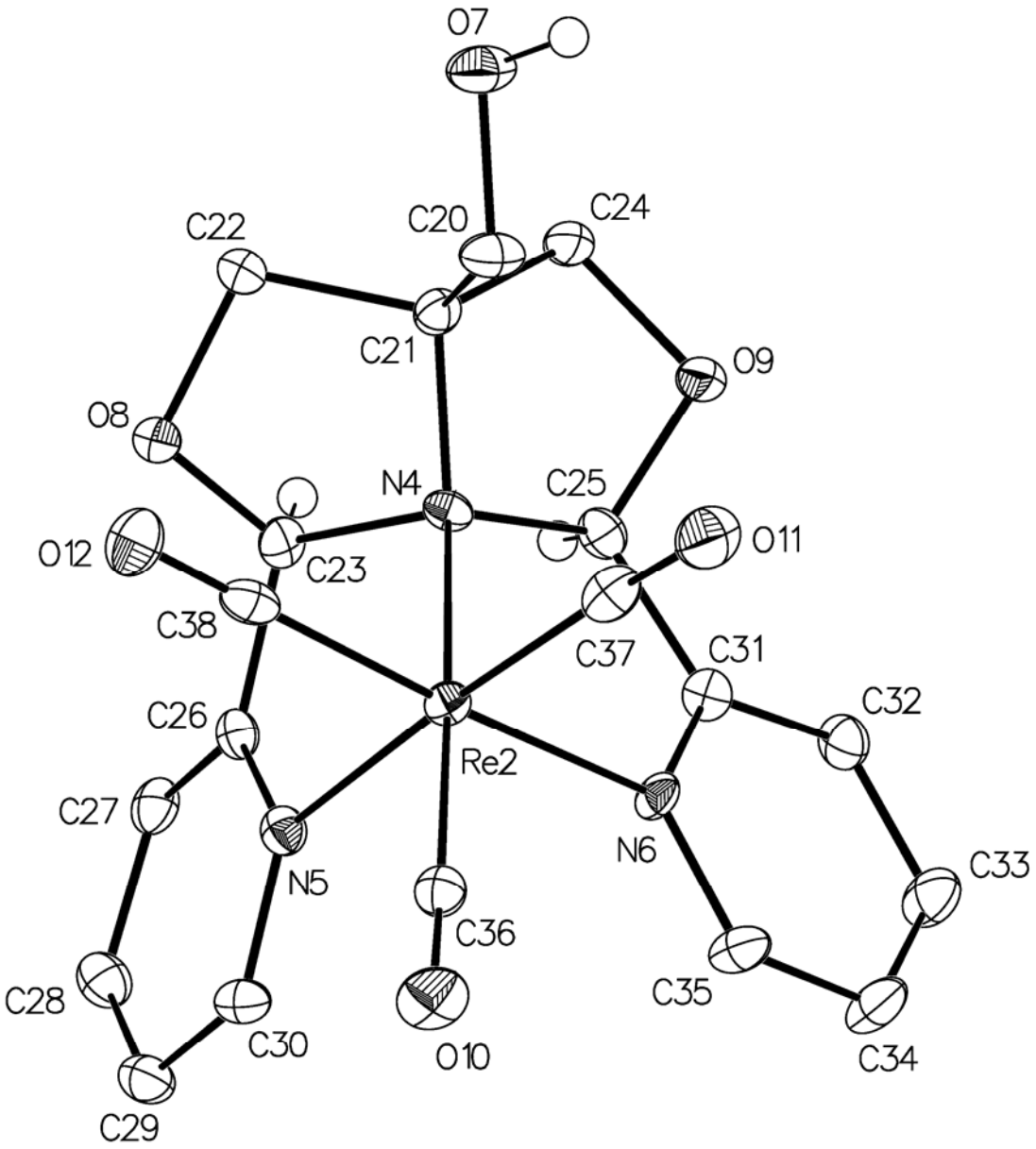
$$P = 1/3 \max (0, F_o^2) + 2/3 F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters




O1
C3
C5
C2
C1
O2
O3
N1
C4
C6
O6
O5
C19
C18
C8
C7
C12
C13
Re1
N2
N3
C9
C14
C17
C11
C16
C10
C15
O4


O7
C22
C20
C24
C21
O9
O8
N4
C25
O12
C23
O11
C38
C37
C31
C26
C32
Re2
C27
N5
N6
C33
C36
C28
C35
C30
O10
C34
C29

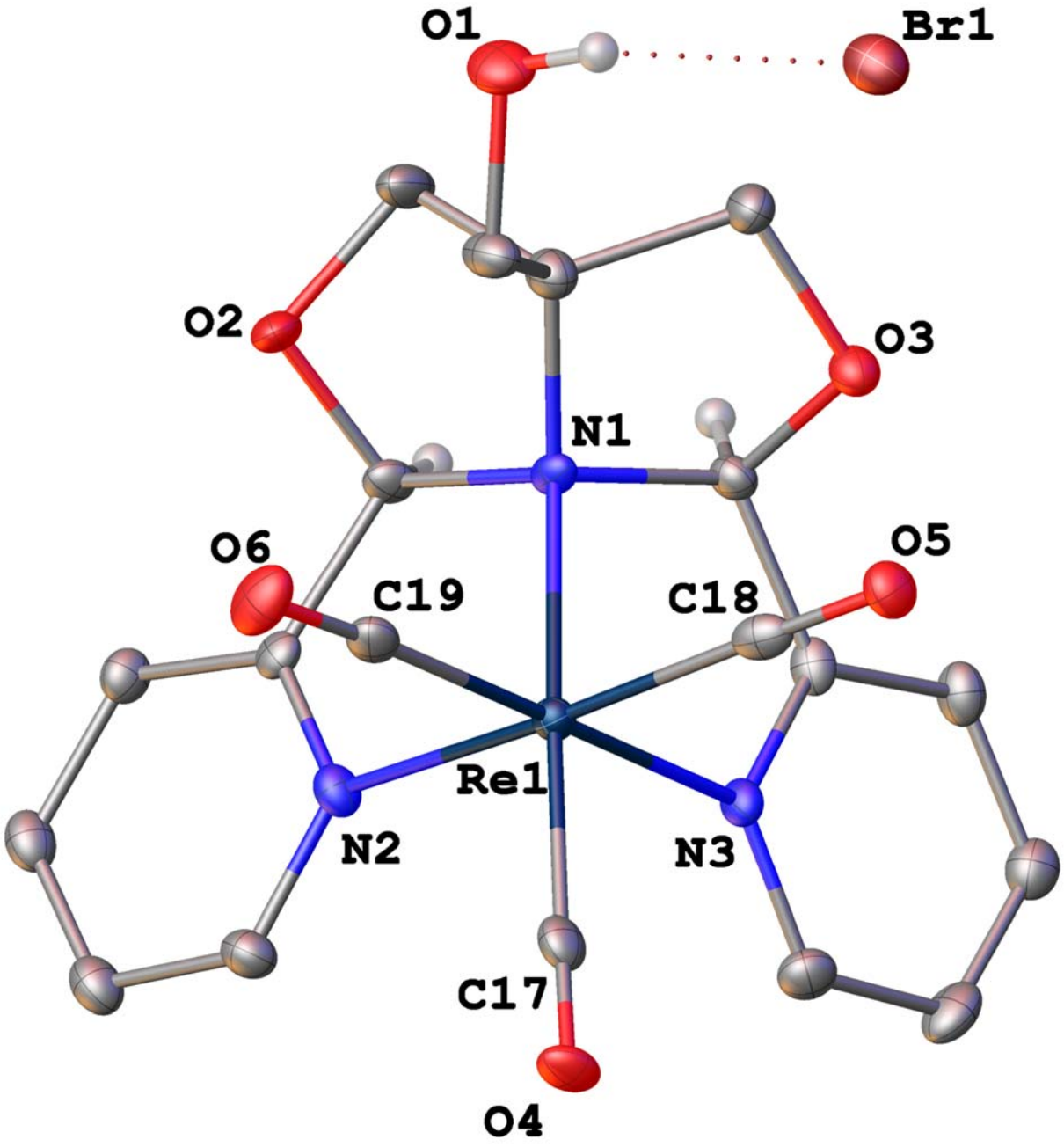

O1
Br1
O2
O3
N1
O6
C19
C18
O5
Re1
N2
N3
C17
O4

Table 1. Crystal data and structure refinement for **8**.

Identification code	jonrap04	
Empirical formula	C19 H17 Br N3 O6 Re	
Formula weight	649.46	
Temperature	100.00(10) K	
Wavelength	1.54184 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 19.2317(2) Å	α = 90°
	b = 10.72530(10) Å	β = 102.0680(10)°
	c = 20.2888(2) Å	γ = 90°
Volume	4092.40(7) Å^3	
Z	8	
Density (calculated)	2.108 Mg/m^3	
Absorption coefficient	14.283 mm^{-1}	
F(000)	2480	
Crystal color, morphology	colourless, block	
Crystal size	0.121 x 0.053 x 0.018 mm^3	
Theta range for data collection	2.880 to 80.217°	
Index ranges	$-24 \le h \le 24$, $-13 \le k \le 13$, $-25 \le l \le 24$	
Reflections collected	53674	
Independent reflections	8801 [R(int) = 0.0489]	
Observed reflections	8381	
Completeness to theta = 74.504°	99.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	1.00000 and 0.84454	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8801 / 0 / 549	
Goodness-of-fit on F^2	1.078	
Final R indices [I>2sigma(I)]	R1 = 0.0286, wR2 = 0.0721	
R indices (all data)	R1 = 0.0304, wR2 = 0.0732	
Largest diff. peak and hole	1.251 and -1.266 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **8**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re1	2318(1)	7494(1)	5345(1)	11(1)
O1	2835(2)	11741(3)	4768(2)	23(1)
O2	4061(2)	8862(3)	4866(2)	19(1)
O3	3178(2)	9773(3)	6554(1)	16(1)
O4	989(2)	5848(3)	5043(2)	18(1)
O5	1405(2)	9344(3)	5958(2)	18(1)
O6	1796(2)	8327(3)	3874(2)	24(1)
N1	3318(2)	8622(3)	5631(2)	11(1)
N2	3091(2)	6273(3)	5036(2)	13(1)
N3	2763(2)	6843(3)	6357(2)	12(1)
C1	2749(2)	10451(4)	4876(2)	18(1)
C2	3361(2)	10010(4)	5442(2)	17(1)
C3	4055(2)	10047(4)	5188(2)	21(1)
C4	3911(2)	8023(4)	5351(2)	14(1)
C5	3357(2)	10700(4)	6103(2)	16(1)
C6	3507(2)	8682(4)	6394(2)	13(1)
C7	3730(2)	6776(4)	5023(2)	12(1)
C8	4239(2)	6147(4)	4757(2)	15(1)
C9	4093(2)	4958(4)	4497(2)	18(1)
C10	3444(2)	4408(4)	4539(2)	18(1)
C11	2959(2)	5095(4)	4807(2)	17(1)
C12	3246(2)	7592(4)	6739(2)	14(1)
C13	3469(2)	7426(4)	7429(2)	17(1)
C14	3208(2)	6424(4)	7729(2)	18(1)
C15	2748(2)	5599(4)	7332(2)	21(1)
C16	2535(2)	5837(4)	6649(2)	17(1)
C17	1503(2)	6411(4)	5166(2)	16(1)
C18	1738(2)	8662(4)	5717(2)	15(1)

C19	1988(2)	8054(4)	4424(2)	15(1)
Re2	5044(1)	3665(1)	7446(1)	12(1)
O7	5004(2)	-801(3)	7802(2)	23(1)
O8	4749(2)	2342(3)	8944(1)	15(1)
O9	6617(2)	1336(3)	8278(2)	20(1)
O10	4337(2)	5553(3)	6383(2)	25(1)
O11	5093(2)	2022(3)	6216(2)	28(1)
O12	3530(2)	2771(3)	7471(2)	26(1)
N4	5601(2)	2480(3)	8309(2)	11(1)
N5	5097(2)	4883(3)	8304(2)	14(1)
N6	6158(2)	4188(3)	7568(2)	13(1)
C20	5098(2)	489(4)	7684(2)	19(1)
C21	5442(2)	1082(4)	8357(2)	14(1)
C22	4936(2)	1053(4)	8858(2)	18(1)
C23	5386(2)	2970(4)	8937(2)	14(1)
C24	6189(2)	547(4)	8611(2)	19(1)
C25	6400(2)	2541(4)	8415(2)	15(1)
C26	5313(2)	4362(4)	8915(2)	13(1)
C27	5471(2)	5063(4)	9499(2)	17(1)
C28	5340(2)	6334(4)	9460(2)	18(1)
C29	5099(2)	6878(4)	8833(2)	18(1)
C30	5013(2)	6123(4)	8267(2)	16(1)
C31	6654(2)	3516(4)	7994(2)	15(1)
C32	7379(2)	3765(4)	8085(2)	17(1)
C33	7599(2)	4730(4)	7724(2)	22(1)
C34	7091(2)	5449(4)	7303(2)	23(1)
C35	6381(2)	5148(4)	7239(2)	18(1)
C36	4614(2)	4869(4)	6781(2)	16(1)
C37	5083(2)	2599(4)	6686(2)	19(1)
C38	4087(2)	3106(4)	7459(2)	16(1)
Br1	1673(1)	12892(1)	5518(1)	22(1)
Br2	5635(1)	-1884(1)	6574(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for **8**.

Re(1)-N(1)	2.245(3)	C(5)-H(5B)	0.9900
Re(1)-N(2)	2.171(3)	C(6)-H(6)	1.0000
Re(1)-N(3)	2.169(3)	C(6)-C(12)	1.502(5)
Re(1)-C(17)	1.923(4)	C(7)-C(8)	1.388(5)
Re(1)-C(18)	1.933(4)	C(8)-H(8)	0.9500
Re(1)-C(19)	1.938(4)	C(8)-C(9)	1.386(6)
O(1)-H(1)	0.86(8)	C(9)-H(9)	0.9500
O(1)-C(1)	1.416(5)	C(9)-C(10)	1.399(6)
O(2)-C(3)	1.430(5)	C(10)-H(10)	0.9500
O(2)-C(4)	1.407(5)	C(10)-C(11)	1.386(6)
O(3)-C(5)	1.441(5)	C(11)-H(11)	0.9500
O(3)-C(6)	1.401(5)	C(12)-C(13)	1.388(6)
O(4)-C(17)	1.141(5)	C(13)-H(13)	0.9500
O(5)-C(18)	1.146(5)	C(13)-C(14)	1.380(6)
O(6)-C(19)	1.137(5)	C(14)-H(14)	0.9500
N(1)-C(2)	1.544(5)	C(14)-C(15)	1.383(7)
N(1)-C(4)	1.518(5)	C(15)-H(15)	0.9500
N(1)-C(6)	1.516(5)	C(15)-C(16)	1.384(6)
N(2)-C(7)	1.346(5)	C(16)-H(16)	0.9500
N(2)-C(11)	1.351(5)	Re(2)-N(4)	2.248(3)
N(3)-C(12)	1.344(5)	Re(2)-N(5)	2.162(3)
N(3)-C(16)	1.347(5)	Re(2)-N(6)	2.179(3)
C(1)-H(1A)	0.9900	Re(2)-C(36)	1.924(4)
C(1)-H(1B)	0.9900	Re(2)-C(37)	1.933(4)
C(1)-C(2)	1.537(6)	Re(2)-C(38)	1.941(4)
C(2)-C(3)	1.528(6)	O(7)-H(7)	0.77(6)
C(2)-C(5)	1.534(5)	O(7)-C(20)	1.422(5)
C(3)-H(3A)	0.9900	O(8)-C(22)	1.448(5)
C(3)-H(3B)	0.9900	O(8)-C(23)	1.399(5)
C(4)-H(4)	1.0000	O(9)-C(24)	1.444(5)
C(4)-C(7)	1.502(5)	O(9)-C(25)	1.403(5)
C(5)-H(5A)	0.9900	O(10)-C(36)	1.137(5)

O(11)-C(37)	1.141(6)	C(33)-C(34)	1.390(6)
O(12)-C(38)	1.133(5)	C(34)-H(34)	0.9500
N(4)-C(21)	1.538(5)	C(34)-C(35)	1.383(6)
N(4)-C(23)	1.514(5)	C(35)-H(35)	0.9500
N(4)-C(25)	1.508(5)	N(2)-Re(1)-N(1)	78.31(12)
N(5)-C(26)	1.344(5)	N(3)-Re(1)-N(1)	77.33(12)
N(5)-C(30)	1.341(5)	N(3)-Re(1)-N(2)	85.42(12)
N(6)-C(31)	1.353(5)	C(17)-Re(1)-N(1)	174.35(15)
N(6)-C(35)	1.344(5)	C(17)-Re(1)-N(2)	99.31(15)
C(20)-H(20A)	0.9900	C(17)-Re(1)-N(3)	97.44(15)
C(20)-H(20B)	0.9900	C(17)-Re(1)-C(18)	87.22(16)
C(20)-C(21)	1.526(6)	C(17)-Re(1)-C(19)	85.15(17)
C(21)-C(22)	1.546(5)	C(18)-Re(1)-N(1)	94.75(14)
C(21)-C(24)	1.532(6)	C(18)-Re(1)-N(2)	171.94(15)
C(22)-H(22A)	0.9900	C(18)-Re(1)-N(3)	89.08(14)
C(22)-H(22B)	0.9900	C(18)-Re(1)-C(19)	94.41(17)
C(23)-H(23)	1.0000	C(19)-Re(1)-N(1)	99.95(14)
C(23)-C(26)	1.500(6)	C(19)-Re(1)-N(2)	90.86(15)
C(24)-H(24A)	0.9900	C(19)-Re(1)-N(3)	175.76(15)
C(24)-H(24B)	0.9900	C(1)-O(1)-H(1)	108(5)
C(25)-H(25)	1.0000	C(4)-O(2)-C(3)	103.0(3)
C(25)-C(31)	1.495(6)	C(6)-O(3)-C(5)	104.4(3)
C(26)-C(27)	1.382(6)	C(2)-N(1)-Re(1)	122.9(2)
C(27)-H(27)	0.9500	C(4)-N(1)-Re(1)	110.2(2)
C(27)-C(28)	1.386(6)	C(4)-N(1)-C(2)	103.4(3)
C(28)-H(28)	0.9500	C(6)-N(1)-Re(1)	107.3(2)
C(28)-C(29)	1.387(6)	C(6)-N(1)-C(2)	101.5(3)
C(29)-H(29)	0.9500	C(6)-N(1)-C(4)	111.2(3)
C(29)-C(30)	1.387(6)	C(7)-N(2)-Re(1)	116.5(3)
C(30)-H(30)	0.9500	C(7)-N(2)-C(11)	118.4(3)
C(31)-C(32)	1.394(6)	C(11)-N(2)-Re(1)	124.8(3)
C(32)-H(32)	0.9500	C(12)-N(3)-Re(1)	116.3(3)
C(32)-C(33)	1.384(6)	C(12)-N(3)-C(16)	118.3(3)
C(33)-H(33)	0.9500	C(16)-N(3)-Re(1)	125.0(3)

O(1)-C(1)-H(1A)	110.0	C(12)-C(6)-N(1)	114.3(3)
O(1)-C(1)-H(1B)	110.0	C(12)-C(6)-H(6)	109.6
O(1)-C(1)-C(2)	108.7(3)	N(2)-C(7)-C(4)	118.5(3)
H(1A)-C(1)-H(1B)	108.3	N(2)-C(7)-C(8)	122.2(4)
C(2)-C(1)-H(1A)	110.0	C(8)-C(7)-C(4)	119.3(3)
C(2)-C(1)-H(1B)	110.0	C(7)-C(8)-H(8)	120.2
C(1)-C(2)-N(1)	114.2(3)	C(9)-C(8)-C(7)	119.5(4)
C(3)-C(2)-N(1)	101.8(3)	C(9)-C(8)-H(8)	120.2
C(3)-C(2)-C(1)	109.1(3)	C(8)-C(9)-H(9)	120.8
C(3)-C(2)-C(5)	116.5(4)	C(8)-C(9)-C(10)	118.5(4)
C(5)-C(2)-N(1)	103.7(3)	C(10)-C(9)-H(9)	120.8
C(5)-C(2)-C(1)	111.2(3)	C(9)-C(10)-H(10)	120.6
O(2)-C(3)-C(2)	102.8(3)	C(11)-C(10)-C(9)	118.8(4)
O(2)-C(3)-H(3A)	111.2	C(11)-C(10)-H(10)	120.6
O(2)-C(3)-H(3B)	111.2	N(2)-C(11)-C(10)	122.5(4)
C(2)-C(3)-H(3A)	111.2	N(2)-C(11)-H(11)	118.8
C(2)-C(3)-H(3B)	111.2	C(10)-C(11)-H(11)	118.8
H(3A)-C(3)-H(3B)	109.1	N(3)-C(12)-C(6)	117.0(3)
O(2)-C(4)-N(1)	105.7(3)	N(3)-C(12)-C(13)	122.4(4)
O(2)-C(4)-H(4)	109.1	C(13)-C(12)-C(6)	120.5(4)
O(2)-C(4)-C(7)	108.8(3)	C(12)-C(13)-H(13)	120.7
N(1)-C(4)-H(4)	109.1	C(14)-C(13)-C(12)	118.6(4)
C(7)-C(4)-N(1)	114.8(3)	C(14)-C(13)-H(13)	120.7
C(7)-C(4)-H(4)	109.1	C(13)-C(14)-H(14)	120.3
O(3)-C(5)-C(2)	105.6(3)	C(13)-C(14)-C(15)	119.3(4)
O(3)-C(5)-H(5A)	110.6	C(15)-C(14)-H(14)	120.3
O(3)-C(5)-H(5B)	110.6	C(14)-C(15)-H(15)	120.5
C(2)-C(5)-H(5A)	110.6	C(14)-C(15)-C(16)	118.9(4)
C(2)-C(5)-H(5B)	110.6	C(16)-C(15)-H(15)	120.5
H(5A)-C(5)-H(5B)	108.8	N(3)-C(16)-C(15)	122.2(4)
O(3)-C(6)-N(1)	104.4(3)	N(3)-C(16)-H(16)	118.9
O(3)-C(6)-H(6)	109.6	C(15)-C(16)-H(16)	118.9
O(3)-C(6)-C(12)	109.3(3)	O(4)-C(17)-Re(1)	174.7(4)
N(1)-C(6)-H(6)	109.6	O(5)-C(18)-Re(1)	177.9(4)

O(6)-C(19)-Re(1)	176.9(4)	H(20A)-C(20)-H(20B)	108.5
N(5)-Re(2)-N(4)	77.25(12)	C(21)-C(20)-H(20A)	110.2
N(5)-Re(2)-N(6)	82.74(12)	C(21)-C(20)-H(20B)	110.2
N(6)-Re(2)-N(4)	76.08(12)	N(4)-C(21)-C(22)	102.8(3)
C(36)-Re(2)-N(4)	172.29(15)	C(20)-C(21)-N(4)	113.8(3)
C(36)-Re(2)-N(5)	95.72(15)	C(20)-C(21)-C(22)	111.4(3)
C(36)-Re(2)-N(6)	100.09(15)	C(20)-C(21)-C(24)	110.2(3)
C(36)-Re(2)-C(37)	85.47(18)	C(24)-C(21)-N(4)	101.6(3)
C(36)-Re(2)-C(38)	87.00(17)	C(24)-C(21)-C(22)	116.5(3)
C(37)-Re(2)-N(4)	101.27(15)	O(8)-C(22)-C(21)	105.4(3)
C(37)-Re(2)-N(5)	175.10(15)	O(8)-C(22)-H(22A)	110.7
C(37)-Re(2)-N(6)	92.38(16)	O(8)-C(22)-H(22B)	110.7
C(37)-Re(2)-C(38)	91.37(18)	C(21)-C(22)-H(22A)	110.7
C(38)-Re(2)-N(4)	96.50(14)	C(21)-C(22)-H(22B)	110.7
C(38)-Re(2)-N(5)	93.44(15)	H(22A)-C(22)-H(22B)	108.8
C(38)-Re(2)-N(6)	172.21(15)	O(8)-C(23)-N(4)	103.6(3)
C(20)-O(7)-H(7)	106(4)	O(8)-C(23)-H(23)	109.4
C(23)-O(8)-C(22)	102.6(3)	O(8)-C(23)-C(26)	113.8(3)
C(25)-O(9)-C(24)	103.0(3)	N(4)-C(23)-H(23)	109.4
C(21)-N(4)-Re(2)	122.1(2)	C(26)-C(23)-N(4)	111.1(3)
C(23)-N(4)-Re(2)	106.8(2)	C(26)-C(23)-H(23)	109.4
C(23)-N(4)-C(21)	101.1(3)	O(9)-C(24)-C(21)	102.1(3)
C(25)-N(4)-Re(2)	113.1(2)	O(9)-C(24)-H(24A)	111.3
C(25)-N(4)-C(21)	104.2(3)	O(9)-C(24)-H(24B)	111.3
C(25)-N(4)-C(23)	108.4(3)	C(21)-C(24)-H(24A)	111.3
C(26)-N(5)-Re(2)	116.4(3)	C(21)-C(24)-H(24B)	111.3
C(30)-N(5)-Re(2)	124.8(3)	H(24A)-C(24)-H(24B)	109.2
C(30)-N(5)-C(26)	118.3(3)	O(9)-C(25)-N(4)	105.5(3)
C(31)-N(6)-Re(2)	118.7(3)	O(9)-C(25)-H(25)	108.4
C(35)-N(6)-Re(2)	123.3(3)	O(9)-C(25)-C(31)	112.7(3)
C(35)-N(6)-C(31)	118.0(3)	N(4)-C(25)-H(25)	108.4
O(7)-C(20)-H(20A)	110.2	C(31)-C(25)-N(4)	113.2(3)
O(7)-C(20)-H(20B)	110.2	C(31)-C(25)-H(25)	108.4
O(7)-C(20)-C(21)	107.5(3)	N(5)-C(26)-C(23)	116.8(3)

N(5)-C(26)-C(27)	122.4(4)	C(32)-C(31)-C(25)	119.7(4)
C(27)-C(26)-C(23)	120.9(4)	C(31)-C(32)-H(32)	120.6
C(26)-C(27)-H(27)	120.6	C(33)-C(32)-C(31)	118.8(4)
C(26)-C(27)-C(28)	118.8(4)	C(33)-C(32)-H(32)	120.6
C(28)-C(27)-H(27)	120.6	C(32)-C(33)-H(33)	120.5
C(27)-C(28)-H(28)	120.4	C(32)-C(33)-C(34)	119.1(4)
C(27)-C(28)-C(29)	119.2(4)	C(34)-C(33)-H(33)	120.5
C(29)-C(28)-H(28)	120.4	C(33)-C(34)-H(34)	120.6
C(28)-C(29)-H(29)	120.9	C(35)-C(34)-C(33)	118.8(4)
C(30)-C(29)-C(28)	118.2(4)	C(35)-C(34)-H(34)	120.6
C(30)-C(29)-H(29)	120.9	N(6)-C(35)-C(34)	122.9(4)
N(5)-C(30)-C(29)	122.7(4)	N(6)-C(35)-H(35)	118.5
N(5)-C(30)-H(30)	118.6	C(34)-C(35)-H(35)	118.5
C(29)-C(30)-H(30)	118.6	O(10)-C(36)-Re(2)	177.4(4)
N(6)-C(31)-C(25)	117.9(3)	O(11)-C(37)-Re(2)	176.4(4)
N(6)-C(31)-C(32)	122.2(4)	O(12)-C(38)-Re(2)	179.4(4)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **8**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Re1	11(1)	11(1)	10(1)	0(1)	1(1)	-1(1)
O1	30(2)	15(2)	22(2)	5(1)	3(1)	1(1)
O2	28(2)	13(1)	20(1)	0(1)	13(1)	-6(1)
O3	20(1)	13(1)	14(1)	1(1)	4(1)	1(1)
O4	12(1)	16(1)	26(2)	-5(1)	3(1)	-6(1)
O5	18(1)	17(1)	19(1)	-2(1)	5(1)	2(1)
O6	21(2)	35(2)	14(1)	6(1)	0(1)	-3(1)
N1	11(2)	13(2)	9(1)	0(1)	-1(1)	-4(1)
N2	14(2)	13(2)	12(2)	-1(1)	1(1)	4(1)
N3	16(2)	12(2)	8(1)	1(1)	1(1)	1(1)
C1	25(2)	14(2)	15(2)	0(2)	1(2)	-4(2)
C2	21(2)	15(2)	14(2)	0(2)	2(2)	-1(2)
C3	24(2)	14(2)	29(2)	-4(2)	14(2)	-5(2)
C4	10(2)	13(2)	18(2)	-1(2)	3(1)	-3(1)
C5	21(2)	15(2)	14(2)	0(2)	5(2)	-2(2)
C6	15(2)	12(2)	12(2)	-2(1)	1(1)	-2(1)
C7	13(2)	14(2)	8(2)	3(1)	1(1)	1(1)
C8	13(2)	17(2)	16(2)	1(2)	4(2)	3(2)
C9	21(2)	17(2)	15(2)	0(2)	3(2)	6(2)
C10	23(2)	15(2)	16(2)	-2(2)	2(2)	4(2)
C11	19(2)	13(2)	17(2)	-1(2)	2(2)	-1(2)
C12	13(2)	16(2)	10(2)	1(1)	-1(1)	5(1)
C13	16(2)	22(2)	12(2)	-1(2)	1(2)	3(2)
C14	19(2)	20(2)	17(2)	2(2)	4(2)	4(2)
C15	24(2)	21(2)	20(2)	10(2)	8(2)	4(2)
C16	14(2)	18(2)	20(2)	1(2)	3(2)	-2(2)
C17	20(2)	13(2)	15(2)	-2(1)	4(2)	4(2)
C18	14(2)	16(2)	13(2)	4(2)	-4(2)	-4(2)

C19	13(2)	16(2)	17(2)	-3(2)	3(2)	-5(2)
Re2	12(1)	12(1)	11(1)	1(1)	1(1)	0(1)
O7	38(2)	14(2)	19(2)	-1(1)	7(1)	-4(1)
O8	17(1)	12(1)	17(1)	0(1)	6(1)	1(1)
O9	17(1)	12(1)	32(2)	4(1)	10(1)	2(1)
O10	25(2)	28(2)	21(2)	11(1)	-1(1)	4(1)
O11	42(2)	24(2)	20(2)	-5(1)	13(1)	-7(2)
O12	14(2)	29(2)	34(2)	-1(1)	5(1)	-5(1)
N4	12(2)	10(2)	11(2)	1(1)	-1(1)	2(1)
N5	12(2)	14(2)	14(2)	-1(1)	2(1)	0(1)
N6	9(1)	15(2)	16(2)	2(1)	3(1)	-3(1)
C20	29(2)	13(2)	15(2)	0(2)	2(2)	-2(2)
C21	14(2)	14(2)	14(2)	3(1)	4(1)	1(2)
C22	20(2)	13(2)	22(2)	2(2)	9(2)	3(2)
C23	16(2)	17(2)	8(2)	2(1)	2(1)	2(2)
C24	16(2)	14(2)	28(2)	6(2)	8(2)	3(2)
C25	12(2)	16(2)	15(2)	1(1)	1(2)	3(1)
C26	11(2)	15(2)	12(2)	-2(1)	2(1)	0(1)
C27	12(2)	20(2)	18(2)	-1(2)	4(2)	-4(2)
C28	19(2)	17(2)	18(2)	-4(2)	3(2)	1(2)
C29	17(2)	14(2)	22(2)	1(2)	3(2)	2(2)
C30	16(2)	12(2)	18(2)	1(2)	1(2)	-1(2)
C31	14(2)	16(2)	13(2)	0(2)	2(1)	0(2)
C32	12(2)	18(2)	21(2)	-1(2)	2(2)	1(2)
C33	15(2)	24(2)	26(2)	3(2)	4(2)	-4(2)
C34	20(2)	23(2)	26(2)	8(2)	7(2)	-6(2)
C35	17(2)	16(2)	21(2)	6(2)	1(2)	-2(2)
C36	15(2)	16(2)	16(2)	-1(2)	3(2)	-1(2)
C37	17(2)	22(2)	17(2)	4(2)	2(2)	-3(2)
C38	21(2)	12(2)	15(2)	1(1)	1(2)	3(2)
Br1	25(1)	19(1)	20(1)	1(1)	0(1)	1(1)
Br2	18(1)	20(1)	28(1)	-3(1)	0(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **8**.

	x	y	z	U(eq)
H1	2530(40)	12140(70)	4950(40)	50(20)
H1A	2754	9981	4457	22
H1B	2287	10301	5005	22
H3A	4054	10738	4865	25
H3B	4471	10139	5566	25
H4	4342	7935	5722	16
H5A	3831	11063	6290	20
H5B	3001	11378	6030	20
H6	4034	8760	6549	16
H8	4683	6528	4752	18
H9	4426	4526	4295	21
H10	3337	3579	4386	22
H11	2516	4724	4831	20
H13	3796	7991	7691	20
H14	3342	6302	8202	22
H15	2582	4881	7526	25
H16	2218	5273	6378	21
H7	5160(30)	-1150(50)	7540(30)	18(14)
H20A	4633	886	7501	23
H20B	5407	599	7355	23
H22A	4507	554	8674	21
H22B	5178	688	9294	21
H23	5751	2724	9343	16
H24A	6336	613	9108	23
H24B	6215	-336	8476	23
H25	6598	2733	8900	18
H27	5665	4680	9919	20
H28	5415	6828	9857	22

H29	4996	7744	8793	22
H30	4889	6502	7835	19
H32	7716	3281	8389	21
H33	8091	4898	7765	26
H34	7229	6135	7063	27
H35	6035	5641	6951	22

Table 6. Torsion angles [°] for **8**.

Re1-N1-C2-C1	-17.2(4)	C2-N1-C6-O3	38.1(4)
Re1-N1-C2-C3	-134.7(3)	C2-N1-C6-C12	157.4(3)
Re1-N1-C2-C5	104.0(3)	C3-O2-C4-N1	43.0(4)
Re1-N1-C4-O2	113.5(3)	C3-O2-C4-C7	166.8(3)
Re1-N1-C4-C7	-6.4(4)	C3-C2-C5-O3	-121.8(4)
Re1-N1-C6-O3	-92.0(3)	C4-O2-C3-C2	-49.0(4)
Re1-N1-C6-C12	27.4(4)	C4-N1-C2-C1	108.0(4)
Re1-N2-C7-C4	12.4(4)	C4-N1-C2-C3	-9.5(4)
Re1-N2-C7-C8	-171.8(3)	C4-N1-C2-C5	-130.8(3)
Re1-N2-C11-C10	171.9(3)	C4-N1-C6-O3	147.5(3)
Re1-N3-C12-C6	-9.4(4)	C4-N1-C6-C12	-93.2(4)
Re1-N3-C12-C13	166.9(3)	C4-C7-C8-C9	175.4(4)
Re1-N3-C16-C15	-167.8(3)	C5-O3-C6-N1	-46.7(4)
O1-C1-C2-N1	177.9(3)	C5-O3-C6-C12	-169.4(3)
O1-C1-C2-C3	-69.0(4)	C5-C2-C3-O2	147.0(4)
O1-C1-C2-C5	60.9(4)	C6-O3-C5-C2	35.7(4)
O2-C4-C7-N2	-121.7(4)	C6-N1-C2-C1	-136.7(3)
O2-C4-C7-C8	62.4(5)	C6-N1-C2-C3	105.9(3)
O3-C6-C12-N3	103.2(4)	C6-N1-C2-C5	-15.5(4)
O3-C6-C12-C13	-73.2(5)	C6-N1-C4-O2	-127.7(3)
N1-C2-C3-O2	35.0(4)	C6-N1-C4-C7	112.4(4)
N1-C2-C5-O3	-10.9(4)	C6-C12-C13-C14	179.0(4)
N1-C4-C7-N2	-3.5(5)	C7-N2-C11-C10	-2.4(6)
N1-C4-C7-C8	-179.4(3)	C7-C8-C9-C10	-2.7(6)
N1-C6-C12-N3	-13.4(5)	C8-C9-C10-C11	3.2(6)
N1-C6-C12-C13	170.2(3)	C9-C10-C11-N2	-0.7(6)
N2-C7-C8-C9	-0.4(6)	C11-N2-C7-C4	-172.9(3)
N3-C12-C13-C14	2.8(6)	C11-N2-C7-C8	2.9(6)
C1-C2-C3-O2	-86.1(4)	C12-N3-C16-C15	4.0(6)
C1-C2-C5-O3	112.3(4)	C12-C13-C14-C15	1.7(6)
C2-N1-C4-O2	-19.5(4)	C13-C14-C15-C16	-3.2(6)
C2-N1-C4-C7	-139.4(3)	C14-C15-C16-N3	0.3(6)

C16-N3-C12-C6	178.1(3)	C21-N4-C25-O9	19.7(4)
C16-N3-C12-C13	-5.6(6)	C21-N4-C25-C31	143.4(3)
Re2-N4-C21-C20	20.8(4)	C22-O8-C23-N4	50.6(3)
Re2-N4-C21-C22	-99.9(3)	C22-O8-C23-C26	171.3(3)
Re2-N4-C21-C24	139.2(3)	C22-C21-C24-O9	-145.8(4)
Re2-N4-C23-O8	86.0(3)	C23-O8-C22-C21	-37.6(4)
Re2-N4-C23-C26	-36.5(3)	C23-N4-C21-C20	138.9(3)
Re2-N4-C25-O9	-115.0(3)	C23-N4-C21-C22	18.2(4)
Re2-N4-C25-C31	8.7(4)	C23-N4-C21-C24	-102.7(3)
Re2-N5-C26-C23	-9.0(4)	C23-N4-C25-O9	126.7(3)
Re2-N5-C26-C27	170.3(3)	C23-N4-C25-C31	-109.6(4)
Re2-N5-C30-C29	-175.4(3)	C23-C26-C27-C28	-174.8(4)
Re2-N6-C31-C25	-6.6(5)	C24-O9-C25-N4	-43.2(4)
Re2-N6-C31-C32	178.0(3)	C24-O9-C25-C31	-167.3(3)
Re2-N6-C35-C34	-177.9(3)	C24-C21-C22-O8	120.4(4)
O7-C20-C21-N4	178.1(3)	C25-O9-C24-C21	49.2(4)
O7-C20-C21-C22	-66.1(4)	C25-N4-C21-C20	-108.7(4)
O7-C20-C21-C24	64.8(4)	C25-N4-C21-C22	130.6(3)
O8-C23-C26-N5	-84.7(4)	C25-N4-C21-C24	9.7(4)
O8-C23-C26-C27	96.0(4)	C25-N4-C23-O8	-151.8(3)
O9-C25-C31-N6	117.9(4)	C25-N4-C23-C26	85.6(4)
O9-C25-C31-C32	-66.5(5)	C25-C31-C32-C33	-175.6(4)
N4-C21-C22-O8	10.4(4)	C26-N5-C30-C29	-3.9(6)
N4-C21-C24-O9	-35.0(4)	C26-C27-C28-C29	-4.1(6)
N4-C23-C26-N5	31.8(5)	C27-C28-C29-C30	-1.4(6)
N4-C23-C26-C27	-147.5(4)	C28-C29-C30-N5	5.6(6)
N4-C25-C31-N6	-1.8(5)	C30-N5-C26-C23	178.7(3)
N4-C25-C31-C32	173.8(4)	C30-N5-C26-C27	-2.0(6)
N5-C26-C27-C28	5.9(6)	C31-N6-C35-C34	2.2(6)
N6-C31-C32-C33	-0.2(6)	C31-C32-C33-C34	2.4(7)
C20-C21-C22-O8	-112.0(4)	C32-C33-C34-C35	-2.3(7)
C20-C21-C24-O9	86.0(4)	C33-C34-C35-N6	0.0(7)
C21-N4-C23-O8	-42.7(3)	C35-N6-C31-C25	173.4(4)
C21-N4-C23-C26	-165.2(3)	C35-N6-C31-C32	-2.1(6)

Table 7. Hydrogen bonds and close contacts for **8** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O1-H1...Br1	0.86(8)	2.35(8)	3.204(4)	170(7)
O7-H7...Br2	0.77(6)	2.45(6)	3.208(4)	169(5)

REFERENCE NUMBER: jonrap11

CRYSTAL STRUCTURE REPORT

$C_{23}\ H_{24}\ N_3\ O_7\ Re$

or

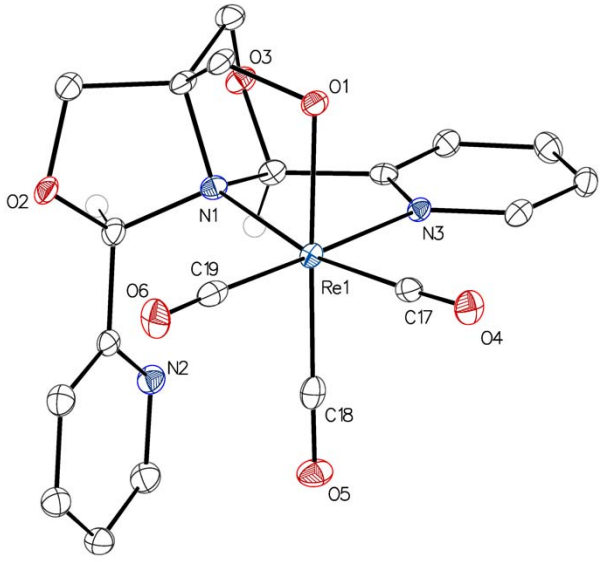
$(\kappa^3\text{-}N,N',O\text{-}L1^{meso\text{-}H})Re(CO)_3 \cdot thf$

(**10**)

Report prepared for:

R. Pohorenec, Prof. W. Jones

September 27, 2022



William W. Brennessel

X-ray Crystallographic Facility

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Rochester, NY 14627

Data collection

A crystal (0.145 x 0.118 x 0.038 mm^3) was placed onto a thin glass optical fiber or a nylon loop and mounted on a Rigaku XtaLAB Synergy-S Dualflex diffractometer equipped with a HyPix-6000HE HPC area detector for data collection at 100.01(10) K. A preliminary set of cell constants and an orientation matrix were calculated from a small sampling of reflections.[1] A short pre-experiment was run, from which an optimal data collection strategy was determined. The full data collection was carried out using a PhotonJet (Cu) X-ray source with a frame time of 0.05 seconds and a detector distance of 34.0 mm. Series of frames were collected in 0.50º steps in ω at different 2θ, κ, and ϕ settings. After the intensity data were corrected for absorption, the final cell constants were calculated from the xyz centroids of 26456 strong reflections from the actual data collection after integration.[1] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT[2] and refined using SHELXL.[3] The space group $P2_1/n$ was determined based on systematic absences. Most or all non-hydrogen atoms were assigned from the solution. Full-matrix least squares / difference Fourier cycles were performed which located any remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0254$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0584$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one Re complex and one tetrahydrofuran solvent molecule in general positions.

Structure manipulation and figure generation were performed using Olex2[4] Unless noted otherwise all structural diagrams containing anisotropic displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B04 Hutchison Hall, Department of Chemistry, University of Rochester. The instrument was purchased with funding from NSF MRI program grant CHE-1725028. .

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1 *CrysAlisPro*, version 171.42.64a; Rigaku Corporation: Oxford, UK, 2022.

2 Sheldrick, G. M. *SHELXT*, version 2018/2; *Acta. Crystallogr*. **2015**, *A71*, 3-8.

3 Sheldrick, G. M. *SHELXL*, version 2019/2; *Acta. Crystallogr*. **2015**, *C71*, 3-8.

4 Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *Olex2*, version 1.5; *J. Appl. Cryst.* **2009**, *42*, 339-341.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

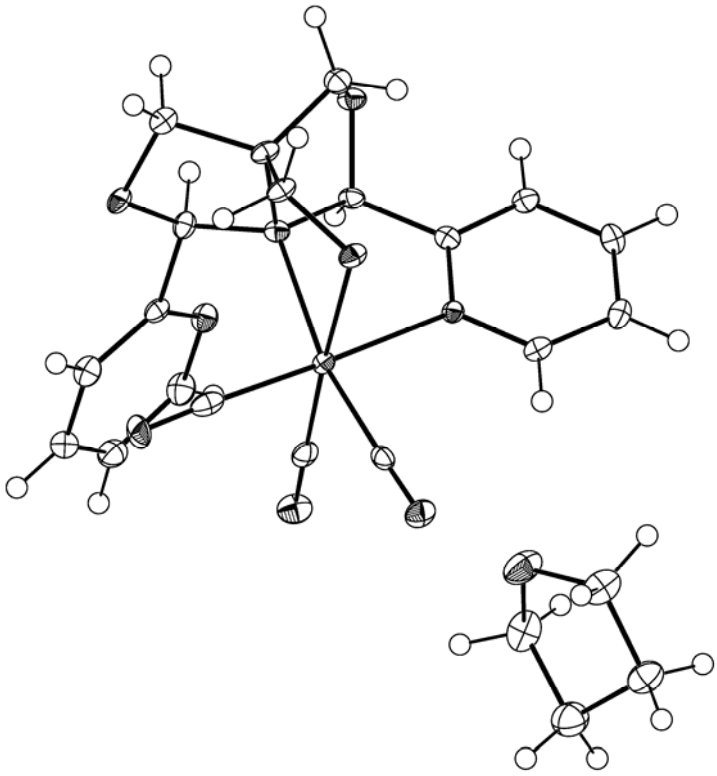
$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

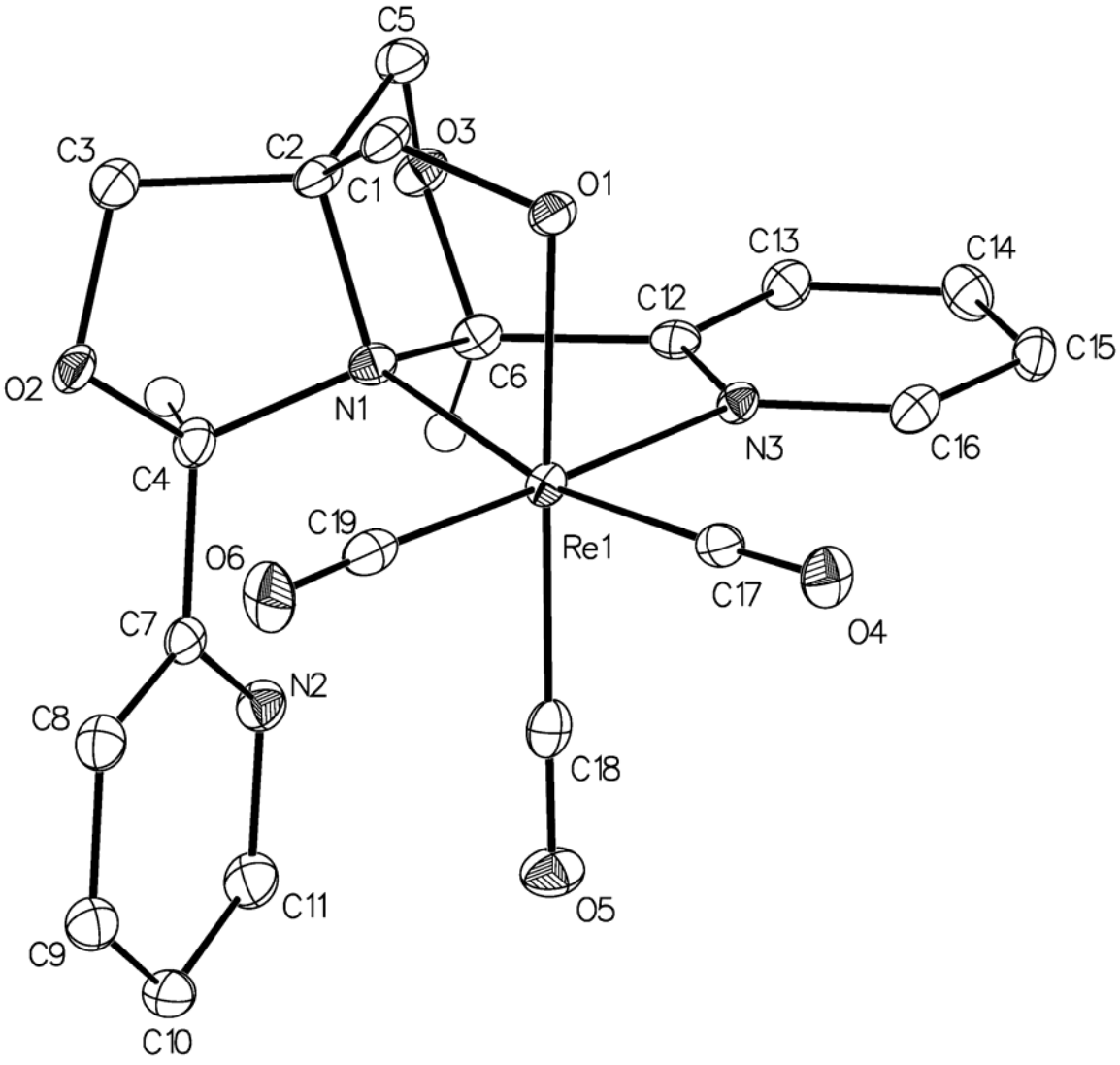
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters




C5
C3
C2
O3
C1
O1
C13
C14
C12
C15
O2
C6
N1
N3
C16
C4
C19
Re1
O6
C17
O4
C7
N2
C8
C18
C11
O5
C9
C10

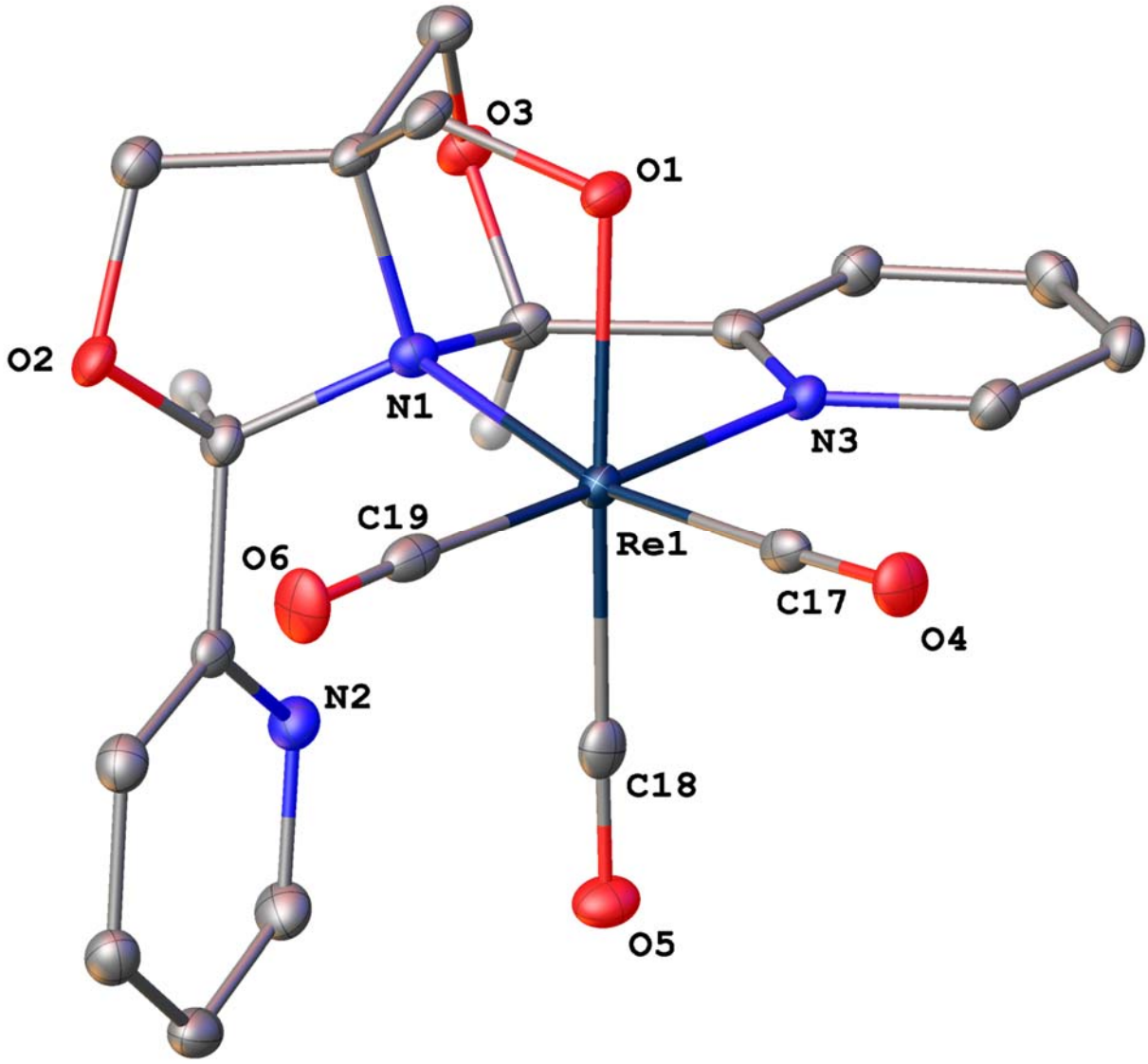

O3
O1
O2
N1
N3
C19
Re1
O6
C17
O4
N2
C18
O5

Table 1. Crystal data and structure refinement for **10**.

Identification code	jonrap11	
Empirical formula	C23 H24 N3 O7 Re	
Formula weight	640.65	
Temperature	100.01(10) K	
Wavelength	1.54184 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 14.08410(10) Å	α = 90°
	b = 10.35030(10) Å	β = 96.0510(10)°
	c = 15.17250(10) Å	γ = 90°
Volume	2199.44(3) Å^3	
Z	4	
Density (calculated)	1.935 Mg/m^3	
Absorption coefficient	11.263 mm^{-1}	
F(000)	1256	
Crystal color, morphology	pale yellow, block	
Crystal size	0.145 x 0.118 x 0.038 mm^3	
Theta range for data collection	4.074 to 80.318°	
Index ranges	$-18 \le h \le 17$, $-11 \le k \le 13$, $-18 \le l \le 19$	
Reflections collected	38647	
Independent reflections	4762 [R(int) = 0.0409]	
Observed reflections	4660	
Completeness to theta = 74.504°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	1.00000 and 0.44515	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4762 / 0 / 307	
Goodness-of-fit on F^2	1.112	
Final R indices [I>2sigma(I)]	R1 = 0.0254, wR2 = 0.0580	
R indices (all data)	R1 = 0.0260, wR2 = 0.0584	
Largest diff. peak and hole	0.619 and -0.935 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **10**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re1	7454(1)	3968(1)	4360(1)	10(1)
O1	8512(2)	3439(2)	3553(2)	14(1)
O2	6391(2)	722(2)	3527(2)	14(1)
O4	7826(2)	6863(2)	4197(2)	18(1)
O5	6033(2)	4586(2)	5714(2)	19(1)
O3	8662(2)	306(2)	5060(2)	14(1)
O6	5786(2)	4324(3)	2905(2)	21(1)
N1	7519(2)	1780(3)	4482(2)	12(1)
N2	5942(2)	1306(3)	5775(2)	15(1)
N3	8632(2)	3671(3)	5388(2)	10(1)
C1	8243(2)	2320(3)	3082(2)	14(1)
C2	8023(2)	1233(3)	3708(2)	13(1)
C3	7286(2)	276(3)	3265(2)	15(1)
C4	6622(2)	934(3)	4435(2)	13(1)
C5	8910(2)	599(3)	4186(2)	15(1)
C6	8139(2)	1399(3)	5298(2)	12(1)
C7	5806(2)	1457(3)	4886(2)	12(1)
C8	4987(2)	1966(3)	4441(2)	16(1)
C9	4255(3)	2345(3)	4928(2)	17(1)
C10	4380(3)	2201(3)	5842(2)	18(1)
C11	5225(3)	1678(4)	6232(2)	18(1)
C12	8801(2)	2463(3)	5675(2)	11(1)
C13	9531(2)	2173(3)	6334(2)	14(1)
C14	10114(2)	3158(3)	6690(2)	17(1)
C15	9961(2)	4408(3)	6382(2)	15(1)
C16	9207(2)	4632(3)	5734(2)	14(1)
C17	7669(2)	5775(3)	4265(2)	13(1)
C18	6555(2)	4347(3)	5213(2)	15(1)

C19	6412(3)	4160(3)	3445(2)	16(1)
O7	7834(2)	6780(3)	6446(2)	29(1)
C20	7306(3)	7068(4)	7175(3)	26(1)
C21	7349(3)	8534(4)	7269(3)	28(1)
C22	8372(3)	8789(4)	7078(2)	23(1)
C23	8558(3)	7744(4)	6409(3)	25(1)

Table 3. Bond lengths [Å] and angles [°] for **10**.

Re(1)-O(1)	2.097(2)	C(6)-H(6)	1.0000
Re(1)-N(1)	2.273(3)	C(6)-C(12)	1.515(4)
Re(1)-N(3)	2.176(3)	C(7)-C(8)	1.378(5)
Re(1)-C(17)	1.903(3)	C(8)-H(8)	0.9500
Re(1)-C(18)	1.945(3)	C(8)-C(9)	1.388(5)
Re(1)-C(19)	1.922(4)	C(9)-H(9)	0.9500
O(1)-C(1)	1.392(4)	C(9)-C(10)	1.388(5)
O(2)-C(3)	1.437(4)	C(10)-H(10)	0.9500
O(2)-C(4)	1.400(4)	C(10)-C(11)	1.383(5)
O(4)-C(17)	1.154(4)	C(11)-H(11)	0.9500
O(5)-C(18)	1.139(4)	C(12)-C(13)	1.390(5)
O(3)-C(5)	1.438(4)	C(13)-H(13)	0.9500
O(3)-C(6)	1.417(4)	C(13)-C(14)	1.383(5)
O(6)-C(19)	1.151(4)	C(14)-H(14)	0.9500
N(1)-C(2)	1.542(4)	C(14)-C(15)	1.385(5)
N(1)-C(4)	1.532(4)	C(15)-H(15)	0.9500
N(1)-C(6)	1.491(4)	C(15)-C(16)	1.388(5)
N(2)-C(7)	1.351(4)	C(16)-H(16)	0.9500
N(2)-C(11)	1.340(4)	O(7)-C(20)	1.429(5)
N(3)-C(12)	1.337(4)	O(7)-C(23)	1.433(5)
N(3)-C(16)	1.353(4)	C(20)-H(20A)	0.9900
C(1)-H(1A)	0.9900	C(20)-H(20B)	0.9900
C(1)-H(1B)	0.9900	C(20)-C(21)	1.524(6)
C(1)-C(2)	1.526(4)	C(21)-H(21A)	0.9900
C(2)-C(3)	1.537(5)	C(21)-H(21B)	0.9900
C(2)-C(5)	1.525(5)	C(21)-C(22)	1.522(6)
C(3)-H(3A)	0.9900	C(22)-H(22A)	0.9900
C(3)-H(3B)	0.9900	C(22)-H(22B)	0.9900
C(4)-H(4)	1.0000	C(22)-C(23)	1.525(5)
C(4)-C(7)	1.499(4)	C(23)-H(23A)	0.9900
C(5)-H(5A)	0.9900	C(23)-H(23B)	0.9900
C(5)-H(5B)	0.9900	O(1)-Re(1)-N(1)	76.30(9)

O(1)-Re(1)-N(3)	81.22(9)	C(1)-C(2)-C(3)	112.0(3)
N(3)-Re(1)-N(1)	77.32(10)	C(3)-C(2)-N(1)	103.0(2)
C(17)-Re(1)-O(1)	94.85(11)	C(5)-C(2)-N(1)	102.5(2)
C(17)-Re(1)-N(1)	168.62(12)	C(5)-C(2)-C(1)	113.8(3)
C(17)-Re(1)-N(3)	94.45(12)	C(5)-C(2)-C(3)	114.4(3)
C(17)-Re(1)-C(18)	88.34(14)	O(2)-C(3)-C(2)	104.1(3)
C(17)-Re(1)-C(19)	87.64(14)	O(2)-C(3)-H(3A)	110.9
C(18)-Re(1)-O(1)	173.54(12)	O(2)-C(3)-H(3B)	110.9
C(18)-Re(1)-N(1)	99.79(12)	C(2)-C(3)-H(3A)	110.9
C(18)-Re(1)-N(3)	92.95(12)	C(2)-C(3)-H(3B)	110.9
C(19)-Re(1)-O(1)	98.12(12)	H(3A)-C(3)-H(3B)	108.9
C(19)-Re(1)-N(1)	100.54(12)	O(2)-C(4)-N(1)	103.9(2)
C(19)-Re(1)-N(3)	177.85(12)	O(2)-C(4)-H(4)	107.7
C(19)-Re(1)-C(18)	87.61(14)	O(2)-C(4)-C(7)	113.3(3)
C(1)-O(1)-Re(1)	110.19(19)	N(1)-C(4)-H(4)	107.7
C(4)-O(2)-C(3)	101.7(2)	C(7)-C(4)-N(1)	116.1(3)
C(6)-O(3)-C(5)	104.5(2)	C(7)-C(4)-H(4)	107.7
C(2)-N(1)-Re(1)	108.74(18)	O(3)-C(5)-C(2)	105.2(2)
C(4)-N(1)-Re(1)	122.71(19)	O(3)-C(5)-H(5A)	110.7
C(4)-N(1)-C(2)	101.5(2)	O(3)-C(5)-H(5B)	110.7
C(6)-N(1)-Re(1)	110.23(19)	C(2)-C(5)-H(5A)	110.7
C(6)-N(1)-C(2)	104.9(2)	C(2)-C(5)-H(5B)	110.7
C(6)-N(1)-C(4)	107.2(2)	H(5A)-C(5)-H(5B)	108.8
C(11)-N(2)-C(7)	116.7(3)	O(3)-C(6)-N(1)	105.9(2)
C(12)-N(3)-Re(1)	117.4(2)	O(3)-C(6)-H(6)	108.6
C(12)-N(3)-C(16)	118.9(3)	O(3)-C(6)-C(12)	111.2(3)
C(16)-N(3)-Re(1)	123.7(2)	N(1)-C(6)-H(6)	108.6
O(1)-C(1)-H(1A)	109.5	N(1)-C(6)-C(12)	113.9(3)
O(1)-C(1)-H(1B)	109.5	C(12)-C(6)-H(6)	108.6
O(1)-C(1)-C(2)	110.8(3)	N(2)-C(7)-C(4)	112.3(3)
H(1A)-C(1)-H(1B)	108.1	N(2)-C(7)-C(8)	123.8(3)
C(2)-C(1)-H(1A)	109.5	C(8)-C(7)-C(4)	123.8(3)
C(2)-C(1)-H(1B)	109.5	C(7)-C(8)-H(8)	120.7
C(1)-C(2)-N(1)	110.1(3)	C(7)-C(8)-C(9)	118.5(3)

C(9)-C(8)-H(8)	120.7	O(6)-C(19)-Re(1)	177.4(3)
C(8)-C(9)-H(9)	120.7	C(20)-O(7)-C(23)	108.4(3)
C(10)-C(9)-C(8)	118.6(3)	O(7)-C(20)-H(20A)	110.7
C(10)-C(9)-H(9)	120.7	O(7)-C(20)-H(20B)	110.7
C(9)-C(10)-H(10)	120.6	O(7)-C(20)-C(21)	105.1(3)
C(11)-C(10)-C(9)	118.9(3)	H(20A)-C(20)-H(20B)	108.8
C(11)-C(10)-H(10)	120.6	C(21)-C(20)-H(20A)	110.7
N(2)-C(11)-C(10)	123.5(3)	C(21)-C(20)-H(20B)	110.7
N(2)-C(11)-H(11)	118.2	C(20)-C(21)-H(21A)	111.6
C(10)-C(11)-H(11)	118.2	C(20)-C(21)-H(21B)	111.6
N(3)-C(12)-C(6)	118.4(3)	H(21A)-C(21)-H(21B)	109.4
N(3)-C(12)-C(13)	121.9(3)	C(22)-C(21)-C(20)	100.7(3)
C(13)-C(12)-C(6)	119.6(3)	C(22)-C(21)-H(21A)	111.6
C(12)-C(13)-H(13)	120.5	C(22)-C(21)-H(21B)	111.6
C(14)-C(13)-C(12)	119.1(3)	C(21)-C(22)-H(22A)	111.0
C(14)-C(13)-H(13)	120.5	C(21)-C(22)-H(22B)	111.0
C(13)-C(14)-H(14)	120.3	C(21)-C(22)-C(23)	103.7(3)
C(13)-C(14)-C(15)	119.5(3)	H(22A)-C(22)-H(22B)	109.0
C(15)-C(14)-H(14)	120.3	C(23)-C(22)-H(22A)	111.0
C(14)-C(15)-H(15)	120.8	C(23)-C(22)-H(22B)	111.0
C(14)-C(15)-C(16)	118.3(3)	O(7)-C(23)-C(22)	107.2(3)
C(16)-C(15)-H(15)	120.8	O(7)-C(23)-H(23A)	110.3
N(3)-C(16)-C(15)	122.3(3)	O(7)-C(23)-H(23B)	110.3
N(3)-C(16)-H(16)	118.8	C(22)-C(23)-H(23A)	110.3
C(15)-C(16)-H(16)	118.8	C(22)-C(23)-H(23B)	110.3
O(4)-C(17)-Re(1)	177.8(3)	H(23A)-C(23)-H(23B)	108.5
O(5)-C(18)-Re(1)	179.1(3)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **10**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Re1	12(1)	9(1)	8(1)	0(1)	1(1)	0(1)
O1	16(1)	12(1)	14(1)	-2(1)	5(1)	-1(1)
O2	17(1)	14(1)	10(1)	-4(1)	1(1)	-2(1)
O4	22(1)	10(1)	22(1)	3(1)	2(1)	-2(1)
O5	23(1)	20(1)	17(1)	1(1)	9(1)	0(1)
O3	18(1)	12(1)	13(1)	0(1)	1(1)	4(1)
O6	20(1)	22(1)	18(1)	2(1)	-10(1)	1(1)
N1	13(1)	10(1)	11(1)	1(1)	2(1)	1(1)
N2	17(1)	17(1)	12(1)	-1(1)	2(1)	-2(1)
N3	12(1)	11(1)	8(1)	-1(1)	0(1)	1(1)
C1	20(2)	12(2)	12(2)	-3(1)	5(1)	-1(1)
C2	17(2)	10(2)	12(1)	-2(1)	6(1)	-1(1)
C3	18(2)	14(2)	14(2)	-1(1)	3(1)	-1(1)
C4	15(2)	15(2)	10(1)	-2(1)	0(1)	-2(1)
C5	16(2)	17(2)	12(2)	-2(1)	2(1)	2(1)
C6	14(2)	10(1)	11(1)	2(1)	1(1)	3(1)
C7	15(2)	9(1)	13(2)	0(1)	4(1)	-3(1)
C8	19(2)	17(2)	14(2)	0(1)	2(1)	-3(1)
C9	17(2)	16(2)	19(2)	-1(1)	1(1)	1(1)
C10	17(2)	18(2)	22(2)	-4(1)	7(1)	-2(1)
C11	20(2)	22(2)	12(2)	-2(1)	3(1)	-3(1)
C12	12(2)	14(2)	9(1)	1(1)	3(1)	2(1)
C13	16(2)	12(2)	15(2)	0(1)	2(1)	1(1)
C14	14(2)	20(2)	16(2)	1(1)	-2(1)	0(1)
C15	16(2)	17(2)	13(2)	-3(1)	1(1)	-4(1)
C16	18(2)	12(2)	14(2)	-2(1)	3(1)	0(1)
C17	11(2)	17(2)	10(1)	-1(1)	-1(1)	4(1)
C18	19(2)	9(2)	17(2)	1(1)	-1(1)	-2(1)

C19	22(2)	10(2)	17(2)	0(1)	10(1)	-1(1)
O7	33(2)	20(1)	35(2)	-6(1)	13(1)	-7(1)
C20	32(2)	27(2)	20(2)	2(2)	3(2)	-8(2)
C21	28(2)	27(2)	31(2)	-5(2)	7(2)	0(2)
C22	33(2)	17(2)	19(2)	1(1)	3(2)	-1(2)
C23	33(2)	20(2)	22(2)	0(2)	10(2)	-5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **10**.

	x	y	z	U(eq)
H1A	8765	2048	2734	17
H1B	7671	2499	2663	17
H3A	7282	296	2612	18
H3B	7425	-615	3478	18
H4	6810	85	4716	16
H5A	9074	-199	3876	18
H5B	9462	1196	4217	18
H6	7722	1133	5760	14
H8	4926	2055	3814	20
H9	3680	2697	4642	21
H10	3893	2457	6194	22
H11	5302	1578	6858	22
H13	9629	1310	6536	17
H14	10615	2978	7144	20
H15	10363	5096	6608	18
H16	9091	5489	5526	17
H20A	7597	6644	7723	32
H20B	6636	6772	7053	32
H21A	6883	8965	6831	34
H21B	7240	8813	7874	34
H22A	8820	8710	7623	27
H22B	8435	9662	6823	27
H23A	8529	8116	5805	30
H23B	9199	7360	6560	30

Table 6. Torsion angles [°] for **10**.

Re1-O1-C1-C2	-55.5(3)	C4-N1-C2-C1	126.3(3)
Re1-N1-C2-C1	-4.3(3)	C4-N1-C2-C3	6.7(3)
Re1-N1-C2-C3	-123.9(2)	C4-N1-C2-C5	-112.3(3)
Re1-N1-C2-C5	117.1(2)	C4-N1-C6-O3	83.7(3)
Re1-N1-C4-O2	86.0(3)	C4-N1-C6-C12	-153.9(3)
Re1-N1-C4-C7	-39.1(3)	C4-C7-C8-C9	176.0(3)
Re1-N1-C6-O3	-140.56(19)	C5-O3-C6-N1	40.2(3)
Re1-N1-C6-C12	-18.1(3)	C5-O3-C6-C12	-84.0(3)
Re1-N3-C12-C6	-2.7(4)	C5-C2-C3-O2	133.2(3)
Re1-N3-C12-C13	-179.6(2)	C6-O3-C5-C2	-40.6(3)
Re1-N3-C16-C15	-179.1(2)	C6-N1-C2-C1	-122.2(3)
O1-C1-C2-N1	37.6(4)	C6-N1-C2-C3	118.2(3)
O1-C1-C2-C3	151.5(3)	C6-N1-C2-C5	-0.8(3)
O1-C1-C2-C5	-76.8(3)	C6-N1-C4-O2	-145.0(2)
O2-C4-C7-N2	162.0(3)	C6-N1-C4-C7	89.8(3)
O2-C4-C7-C8	-14.5(5)	C6-C12-C13-C14	-178.2(3)
O3-C6-C12-N3	134.1(3)	C7-N2-C11-C10	-0.3(5)
O3-C6-C12-C13	-48.9(4)	C7-C8-C9-C10	0.3(5)
N1-C2-C3-O2	22.8(3)	C8-C9-C10-C11	-0.4(5)
N1-C2-C5-O3	24.6(3)	C9-C10-C11-N2	0.4(6)
N1-C4-C7-N2	-77.8(3)	C11-N2-C7-C4	-176.3(3)
N1-C4-C7-C8	105.7(4)	C11-N2-C7-C8	0.2(5)
N1-C6-C12-N3	14.6(4)	C12-N3-C16-C15	-0.6(5)
N1-C6-C12-C13	-168.5(3)	C12-C13-C14-C15	-0.4(5)
N2-C7-C8-C9	-0.1(5)	C13-C14-C15-C16	1.5(5)
N3-C12-C13-C14	-1.4(5)	C14-C15-C16-N3	-1.1(5)
C1-C2-C3-O2	-95.5(3)	C16-N3-C12-C6	178.7(3)
C1-C2-C5-O3	143.4(3)	C16-N3-C12-C13	1.8(4)
C2-N1-C4-O2	-35.3(3)	O7-C20-C21-C22	-38.7(4)
C2-N1-C4-C7	-160.4(3)	C20-O7-C23-C22	-8.3(4)
C2-N1-C6-O3	-23.6(3)	C20-C21-C22-C23	32.7(4)
C2-N1-C6-C12	98.8(3)	C21-C22-C23-O7	-16.5(4)
C3-O2-C4-N1	51.2(3)	C23-O7-C20-C21	29.9(4)
C3-O2-C4-C7	178.1(3)		
C3-C2-C5-O3	-86.2(3)		
C4-O2-C3-C2	-46.2(3)		

REFERENCE NUMBER: jonrap12

CRYSTAL STRUCTURE REPORT

$C_{19}\ H_{16}\ N_3\ O_6\ Re$

or

$(\kappa^3\text{-}N,N',O\text{-}L1^{meso\text{-}H})Re(CO)_3$

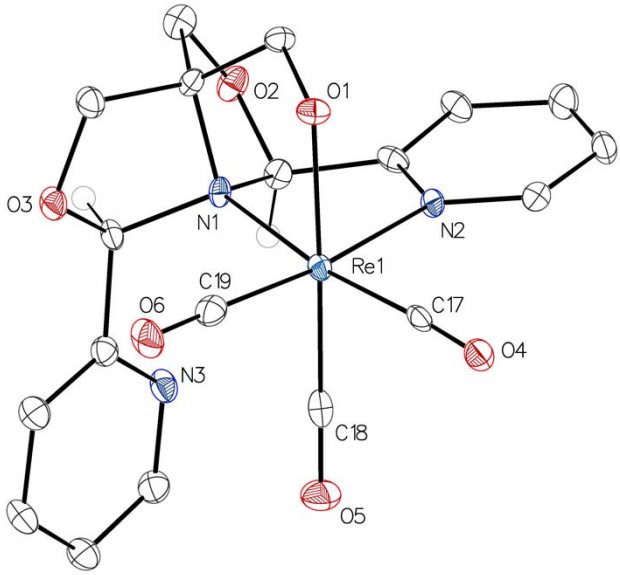
(**10b**)

Second Polymorph

Report prepared for:

R. Pohorenec, Prof. W. Jones

December 16, 2022



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Data collection

A crystal (0.105 x 0.053 x 0.023 mm^3) was placed onto a thin glass optical fiber or a nylon loop and mounted on a Rigaku XtaLAB Synergy-S Dualflex diffractometer equipped with a HyPix-6000HE HPC area detector for data collection at 100.00(10) K. A preliminary set of cell constants and an orientation matrix were calculated from a small sampling of reflections.[1] A short pre-experiment was run, from which an optimal data collection strategy was determined. The full data collection was carried out using a PhotonJet (Cu) X-ray source with frame times of 0.06 and 0.22 seconds and a detector distance of 34.0 mm. Series of frames were collected in 0.50º steps in ω at different 2θ, κ, and ϕ settings. After the intensity data were corrected for absorption, the final cell constants were calculated from the xyz centroids of 37583 strong reflections from the actual data collection after integration.[1] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT[2] and refined using SHELXL.[3] The space group *P*-1 was determined based on intensity statistics. Most or all non-hydrogen atoms were assigned from the solution. Full-matrix least squares / difference Fourier cycles were performed which located any remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0259$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0678$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains two molecules in general positions. The -CH2- (i.e., methylene) region of molecule Re2 is modeled as disordered over two positions (0.82:0.18).

Structure manipulation and figure generation were performed using Olex2.[4] Unless noted otherwise all structural diagrams containing anisotropic displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B04 Hutchison Hall, Department of Chemistry, University of Rochester. The instrument was purchased with funding from NSF MRI program grant CHE-1725028.

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1 *CrysAlisPro*, version 171.42.72a; Rigaku Corporation: Oxford, UK, 2022.

2 Sheldrick, G. M. *SHELXT*, version 2018/2; *Acta. Crystallogr.* **2015**, *A71*, 3-8.

3 Sheldrick, G. M. *SHELXL*, version 2019/2; *Acta. Crystallogr.* **2015**, *C71*, 3-8.

4 Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *Olex2*, version 1.5; *J. Appl. Cryst.* **2009**, *42*, 339-341.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

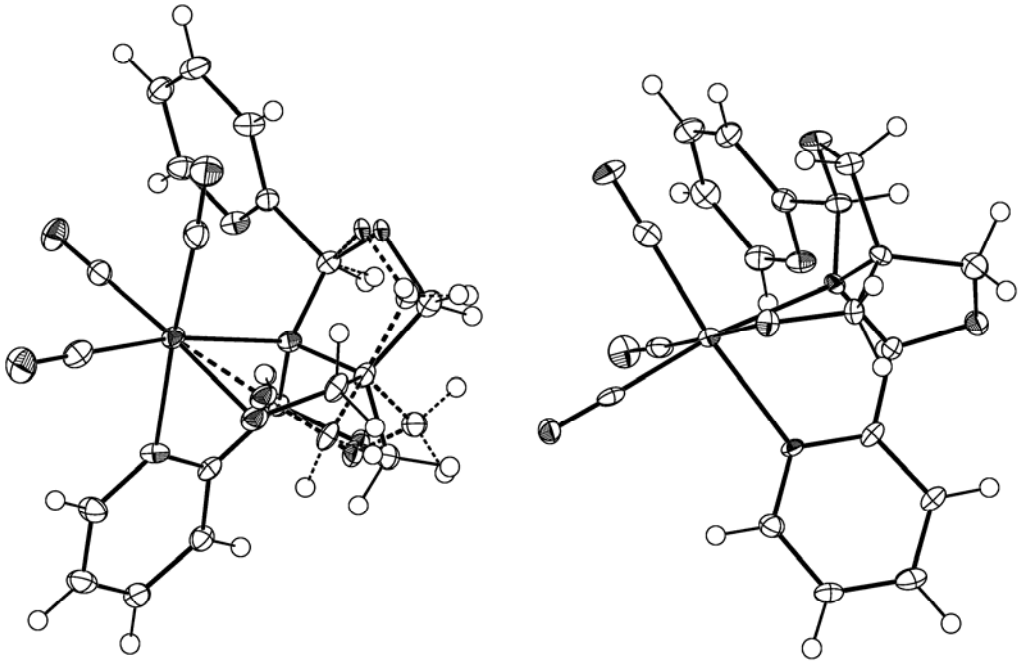
$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

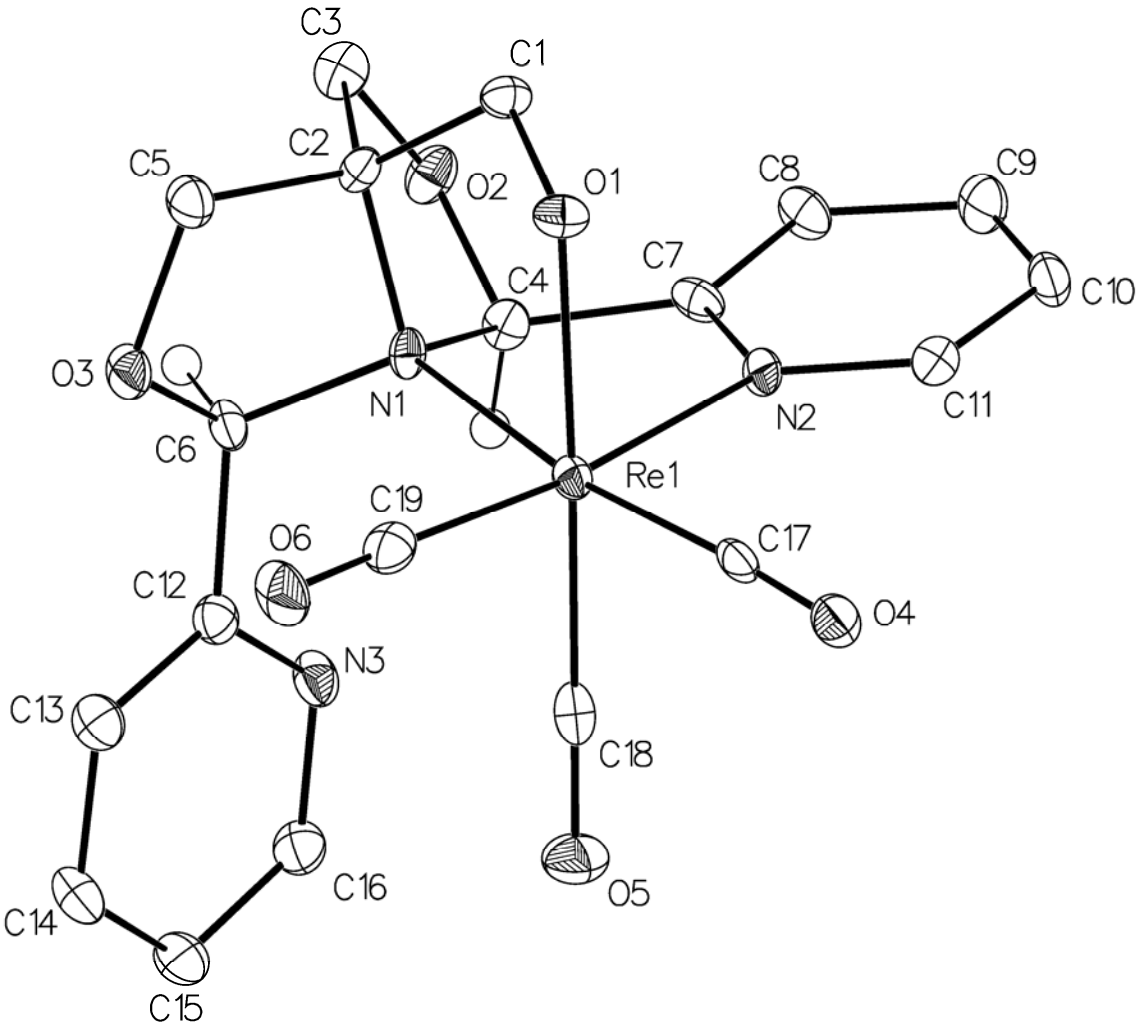
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

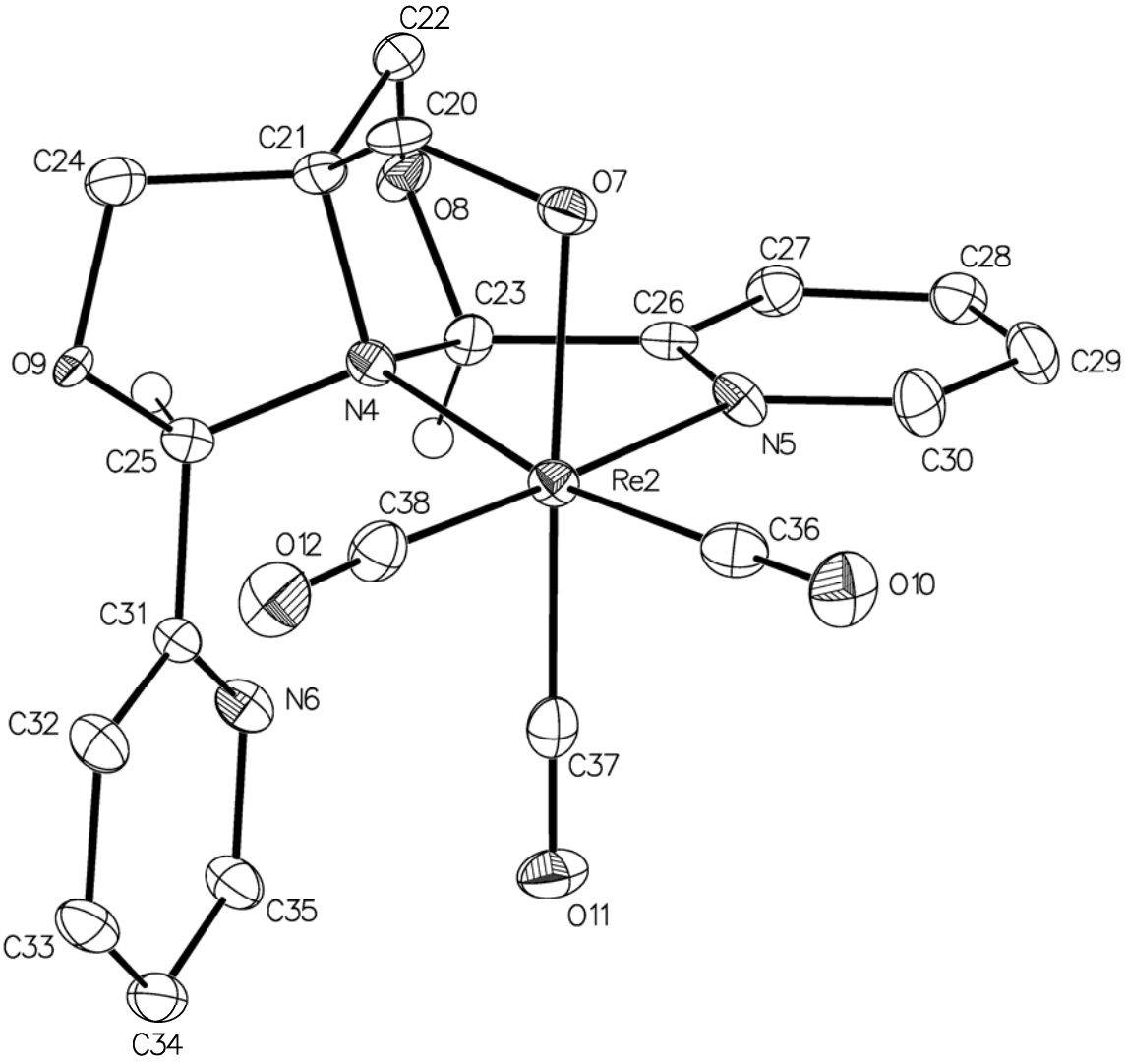
$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters




C3
C1
C2
C5
O2
O1
C8
C9
C7
C4
C10
O3
N1
N2
C11
C6
Re1
C19
O6
C17
C12
O4
N3
C13
C18
C16
O5
C14
C15


C22
C20
C21
C24
O7
O8
C27
C28
C23
C26
O9
C29
N4
N5
C25
C30
Re2
C38
C36
O12
O10
C31
N6
C32
C37
C35
O11
C33
C34

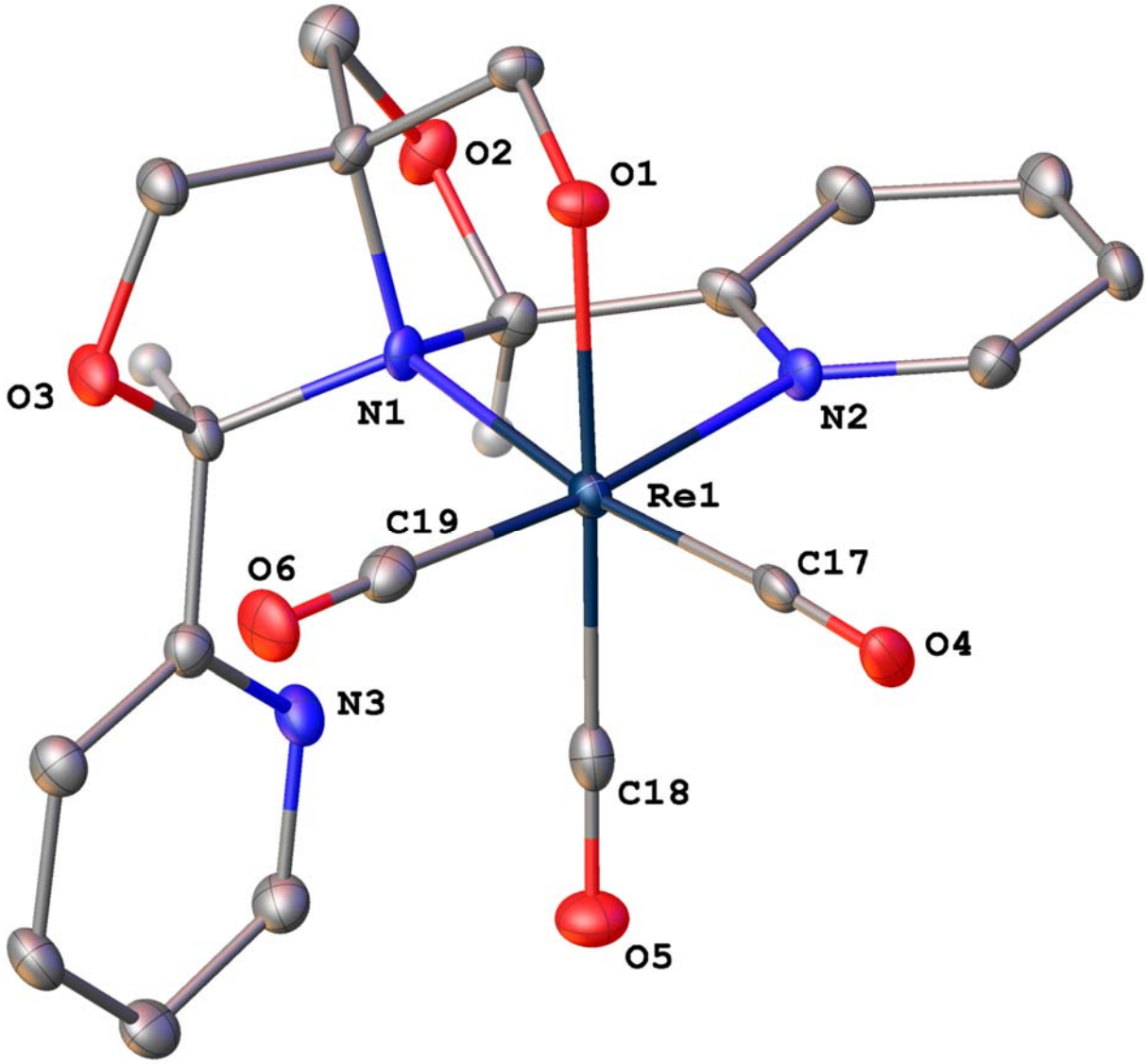

O2
O1
O3
N1
N2
Re1
C19
O6
C17
O4
N3
C18
O5

Table 1. Crystal data and structure refinement for **10**.

Identification code	jonrap12	
Empirical formula	C19 H16 N3 O6 Re	
Formula weight	568.55	
Temperature	100.00(10) K	
Wavelength	1.54184 Å	
Crystal system	triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 10.03030(10) Å	α = 81.8090(10)°
	b = 10.4951(2) Å	β = 81.8630(10)°
	c = 17.6513(2) Å	γ = 81.9800(10)°
Volume	1807.11(4) Å^3	
Z	4	
Density (calculated)	2.090 Mg/m^3	
Absorption coefficient	13.560 mm^{-1}	
F(000)	1096	
Crystal color, morphology	colourless, block	
Crystal size	0.105 x 0.053 x 0.023 mm^3	
Theta range for data collection	4.288 to 80.228°	
Index ranges	$-12 \le h \le 12$, $-13 \le k \le 12$, $-22 \le l \le 22$	
Reflections collected	61205	
Independent reflections	7771 [R(int) = 0.0466]	
Observed reflections	7396	
Completeness to theta = 74.504°	99.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	1.00000 and 0.79636	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7771 / 57 / 578	
Goodness-of-fit on F^2	1.134	
Final R indices [I>2sigma(I)]	R1 = 0.0259, wR2 = 0.0670	
R indices (all data)	R1 = 0.0273, wR2 = 0.0678	
Largest diff. peak and hole	0.913 and -1.316 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **10**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re1	8028(1)	3271(1)	5430(1)	11(1)
Re2	8752(1)	2592(1)	663(1)	13(1)
O7	9672(5)	3848(4)	1180(2)	18(1)
O8	5922(11)	4627(10)	2229(6)	17(2)
O9	7684(4)	1281(4)	2760(2)	18(1)
O10	10739(3)	2686(3)	-823(2)	28(1)
O11	7093(3)	1006(3)	-106(2)	23(1)
O12	10327(3)	-14(3)	1212(2)	31(1)
N4	7321(3)	2897(3)	1747(2)	13(1)
N5	7545(3)	4423(3)	329(2)	16(1)
N6	5045(3)	1190(3)	1602(2)	19(1)
C20	9567(4)	3482(5)	1968(3)	17(1)
C21	8072(4)	3438(4)	2311(2)	15(1)
C22	7287(5)	4766(5)	2391(3)	16(1)
C23	6175(4)	3932(3)	1576(2)	14(1)
C24	7952(6)	2427(5)	3029(3)	20(1)
C25	6714(4)	1786(4)	2275(2)	17(1)
C26	6477(4)	4802(3)	829(2)	14(1)
C27	5601(4)	5921(4)	663(2)	16(1)
C28	5856(4)	6685(4)	-33(2)	19(1)
C29	6992(4)	6326(4)	-542(2)	21(1)
C30	7811(4)	5190(4)	-342(2)	21(1)
C31	6244(4)	790(4)	1875(2)	15(1)
C32	6939(4)	-434(4)	1833(2)	20(1)
C33	6377(4)	-1310(4)	1492(2)	21(1)
C34	5155(4)	-918(4)	1197(2)	19(1)
C35	4536(4)	336(4)	1262(2)	19(1)
C36	10004(4)	2643(4)	-267(2)	20(1)
C37	7704(4)	1601(4)	186(2)	18(1)
C38	9748(4)	976(4)	1023(2)	20(1)

O4	8999(3)	4440(3)	3792(2)	18(1)
O6	8532(3)	609(3)	4845(2)	23(1)
O5	5303(3)	3596(3)	4772(2)	23(1)
O1	9894(2)	2978(3)	5890(2)	15(1)
O2	6956(3)	3441(3)	7868(2)	18(1)
O3	7590(3)	431(2)	6776(2)	18(1)
N1	7367(3)	2647(3)	6675(2)	12(1)
N2	7723(3)	5075(3)	5964(2)	12(1)
N3	4441(3)	2495(3)	6549(2)	16(1)
C12	5393(4)	1467(4)	6480(2)	14(1)
C9	7507(4)	7218(4)	6762(2)	19(1)
C7	7213(4)	5002(4)	6717(2)	14(1)
C14	4023(4)	393(4)	5839(2)	19(1)
C11	8122(4)	6207(4)	5608(2)	16(1)
C17	8677(4)	4003(3)	4417(2)	14(1)
C8	7109(4)	6054(4)	7135(2)	16(1)
C19	8347(4)	1598(4)	5072(2)	17(1)
C10	8013(4)	7299(4)	5987(2)	17(1)
C5	8843(4)	733(4)	6972(2)	16(1)
C6	6599(3)	1486(3)	6899(2)	13(1)
C13	5242(4)	412(4)	6124(2)	17(1)
C1	9775(4)	2914(4)	6684(2)	13(1)
C15	3035(4)	1451(4)	5901(2)	19(1)
C2	8613(4)	2177(3)	7080(2)	13(1)
C18	6292(4)	3498(3)	5049(2)	16(1)
C3	8149(4)	2491(4)	7907(2)	18(1)
C4	6696(4)	3751(3)	7082(2)	14(1)
C16	3303(4)	2478(4)	6241(2)	18(1)
O9A	7910(20)	1229(19)	2582(15)	19(2)
C24A	8530(30)	2238(17)	2829(14)	20(2)
C22A	6888(19)	4340(20)	2706(12)	17(2)
O8A	5940(50)	4780(60)	2150(30)	17(3)
C20A	9098(19)	4253(19)	1819(10)	16(2)
O7A	9890(20)	3510(20)	1292(12)	17(2)

Table 3. Bond lengths [Å] and angles [°] for **10**.

Re(1)-O(1)	2.110(2)	C(21)-C(24)	1.535(6)
Re(1)-N(1)	2.234(3)	C(21)-C(24A)	1.505(16)
Re(1)-N(2)	2.197(3)	C(21)-C(22A)	1.558(15)
Re(1)-C(17)	1.905(4)	C(21)-C(20A)	1.524(15)
Re(1)-C(19)	1.920(4)	C(22)-H(22A)	0.9900
Re(1)-C(18)	1.926(4)	C(22)-H(22B)	0.9900
Re(2)-O(7)	2.083(4)	C(23)-H(23)	1.0000
Re(2)-N(4)	2.256(3)	C(23)-H(23A)	1.0000
Re(2)-N(5)	2.176(3)	C(23)-C(26)	1.511(5)
Re(2)-C(36)	1.921(4)	C(23)-O(8A)	1.412(17)
Re(2)-C(37)	1.925(4)	C(24)-H(24A)	0.9900
Re(2)-C(38)	1.918(4)	C(24)-H(24B)	0.9900
Re(2)-O(7A)	2.106(16)	C(25)-H(25)	1.0000
O(7)-C(20)	1.383(6)	C(25)-H(25A)	1.0000
O(8)-C(22)	1.468(11)	C(25)-C(31)	1.501(5)
O(8)-C(23)	1.424(6)	C(25)-O(9A)	1.409(17)
O(9)-C(24)	1.426(6)	C(26)-C(27)	1.387(5)
O(9)-C(25)	1.387(5)	C(27)-H(27)	0.9500
O(10)-C(36)	1.140(5)	C(27)-C(28)	1.380(6)
O(11)-C(37)	1.148(5)	C(28)-H(28)	0.9500
O(12)-C(38)	1.147(5)	C(28)-C(29)	1.391(6)
N(4)-C(21)	1.540(5)	C(29)-H(29)	0.9500
N(4)-C(23)	1.500(4)	C(29)-C(30)	1.382(6)
N(4)-C(25)	1.525(5)	C(30)-H(30)	0.9500
N(5)-C(26)	1.342(5)	C(31)-C(32)	1.380(5)
N(5)-C(30)	1.349(5)	C(32)-H(32)	0.9500
N(6)-C(31)	1.350(5)	C(32)-C(33)	1.387(6)
N(6)-C(35)	1.338(5)	C(33)-H(33)	0.9500
C(20)-H(20A)	0.9900	C(33)-C(34)	1.387(6)
C(20)-H(20B)	0.9900	C(34)-H(34)	0.9500
C(20)-C(21)	1.539(6)	C(34)-C(35)	1.388(5)
C(21)-C(22)	1.517(6)	C(35)-H(35)	0.9500

O(4)-C(17)	1.153(5)	C(1)-H(1A)	0.9900
O(6)-C(19)	1.148(5)	C(1)-H(1B)	0.9900
O(5)-C(18)	1.153(5)	C(1)-C(2)	1.520(5)
O(1)-C(1)	1.383(4)	C(15)-H(15)	0.9500
O(2)-C(3)	1.449(4)	C(15)-C(16)	1.380(6)
O(2)-C(4)	1.430(4)	C(2)-C(3)	1.535(5)
O(3)-C(5)	1.437(4)	C(3)-H(3A)	0.9900
O(3)-C(6)	1.401(4)	C(3)-H(3B)	0.9900
N(1)-C(6)	1.508(4)	C(4)-H(4)	1.0000
N(1)-C(2)	1.516(4)	C(16)-H(16)	0.9500
N(1)-C(4)	1.482(4)	O(9A)-C(24A)	1.444(17)
N(2)-C(7)	1.351(5)	C(24A)-H(24C)	0.9900
N(2)-C(11)	1.347(5)	C(24A)-H(24D)	0.9900
N(3)-C(12)	1.345(5)	C(22A)-H(22C)	0.9900
N(3)-C(16)	1.335(5)	C(22A)-H(22D)	0.9900
C(12)-C(6)	1.508(5)	C(22A)-O(8A)	1.45(2)
C(12)-C(13)	1.384(5)	C(20A)-H(20C)	0.9900
C(9)-H(9)	0.9500	C(20A)-H(20D)	0.9900
C(9)-C(8)	1.385(6)	C(20A)-O(7A)	1.387(17)
C(9)-C(10)	1.385(6)	O(1)-Re(1)-N(1)	78.60(10)
C(7)-C(8)	1.397(5)	O(1)-Re(1)-N(2)	83.71(10)
C(7)-C(4)	1.504(5)	N(2)-Re(1)-N(1)	75.80(11)
C(14)-H(14)	0.9500	C(17)-Re(1)-O(1)	97.53(13)
C(14)-C(13)	1.390(5)	C(17)-Re(1)-N(1)	172.02(13)
C(14)-C(15)	1.387(6)	C(17)-Re(1)-N(2)	96.93(13)
C(11)-H(11)	0.9500	C(17)-Re(1)-C(19)	88.38(15)
C(11)-C(10)	1.391(5)	C(17)-Re(1)-C(18)	84.83(15)
C(8)-H(8)	0.9500	C(19)-Re(1)-O(1)	92.13(13)
C(10)-H(10)	0.9500	C(19)-Re(1)-N(1)	98.68(14)
C(5)-H(5A)	0.9900	C(19)-Re(1)-N(2)	173.63(14)
C(5)-H(5B)	0.9900	C(19)-Re(1)-C(18)	87.54(15)
C(5)-C(2)	1.537(5)	C(18)-Re(1)-O(1)	177.61(13)
C(6)-H(6)	1.0000	C(18)-Re(1)-N(1)	99.11(13)
C(13)-H(13)	0.9500	C(18)-Re(1)-N(2)	96.41(13)

O(7)-Re(2)-N(4)	76.67(14)	O(7)-C(20)-H(20B)	109.5
O(7)-Re(2)-N(5)	79.76(15)	O(7)-C(20)-C(21)	110.7(4)
N(5)-Re(2)-N(4)	77.38(12)	H(20A)-C(20)-H(20B)	108.1
C(36)-Re(2)-O(7)	95.53(17)	C(21)-C(20)-H(20A)	109.5
C(36)-Re(2)-N(4)	170.15(14)	C(21)-C(20)-H(20B)	109.5
C(36)-Re(2)-N(5)	95.47(15)	N(4)-C(21)-C(22A)	100.5(7)
C(36)-Re(2)-C(37)	87.65(16)	C(20)-C(21)-N(4)	109.3(3)
C(36)-Re(2)-O(7A)	96.4(6)	C(22)-C(21)-N(4)	103.3(3)
C(37)-Re(2)-O(7)	172.63(16)	C(22)-C(21)-C(20)	113.7(4)
C(37)-Re(2)-N(4)	99.42(13)	C(22)-C(21)-C(24)	116.0(4)
C(37)-Re(2)-N(5)	93.34(14)	C(24)-C(21)-N(4)	102.7(3)
C(37)-Re(2)-O(7A)	173.6(7)	C(24)-C(21)-C(20)	110.8(4)
C(38)-Re(2)-O(7)	99.53(17)	C(24A)-C(21)-N(4)	102.6(8)
C(38)-Re(2)-N(4)	98.93(14)	C(24A)-C(21)-C(22A)	113.5(14)
C(38)-Re(2)-N(5)	176.31(14)	C(24A)-C(21)-C(20A)	121.0(13)
C(38)-Re(2)-C(36)	88.19(17)	C(20A)-C(21)-N(4)	106.6(7)
C(38)-Re(2)-C(37)	87.20(17)	C(20A)-C(21)-C(22A)	110.0(12)
C(38)-Re(2)-O(7A)	87.9(7)	O(8)-C(22)-C(21)	103.8(4)
O(7A)-Re(2)-N(4)	77.2(6)	O(8)-C(22)-H(22A)	111.0
O(7A)-Re(2)-N(5)	91.3(6)	O(8)-C(22)-H(22B)	111.0
C(20)-O(7)-Re(2)	109.8(3)	C(21)-C(22)-H(22A)	111.0
C(23)-O(8)-C(22)	103.6(7)	C(21)-C(22)-H(22B)	111.0
C(25)-O(9)-C(24)	101.2(4)	H(22A)-C(22)-H(22B)	109.0
C(21)-N(4)-Re(2)	108.9(2)	O(8)-C(23)-N(4)	105.0(3)
C(23)-N(4)-Re(2)	110.5(2)	O(8)-C(23)-H(23)	108.6
C(23)-N(4)-C(21)	104.8(3)	O(8)-C(23)-C(26)	112.5(6)
C(23)-N(4)-C(25)	107.4(3)	N(4)-C(23)-H(23)	108.6
C(25)-N(4)-Re(2)	122.7(2)	N(4)-C(23)-H(23A)	109.7
C(25)-N(4)-C(21)	100.8(3)	N(4)-C(23)-C(26)	113.3(3)
C(26)-N(5)-Re(2)	117.1(3)	C(26)-C(23)-H(23)	108.6
C(26)-N(5)-C(30)	118.7(3)	C(26)-C(23)-H(23A)	109.7
C(30)-N(5)-Re(2)	124.1(3)	O(8A)-C(23)-N(4)	110.0(10)
C(35)-N(6)-C(31)	116.7(3)	O(8A)-C(23)-H(23A)	109.7
O(7)-C(20)-H(20A)	109.5	O(8A)-C(23)-C(26)	104(3)

O(9)-C(24)-C(21)	105.4(4)	C(32)-C(31)-C(25)	122.9(3)
O(9)-C(24)-H(24A)	110.7	C(31)-C(32)-H(32)	120.8
O(9)-C(24)-H(24B)	110.7	C(31)-C(32)-C(33)	118.5(4)
C(21)-C(24)-H(24A)	110.7	C(33)-C(32)-H(32)	120.8
C(21)-C(24)-H(24B)	110.7	C(32)-C(33)-H(33)	120.5
H(24A)-C(24)-H(24B)	108.8	C(32)-C(33)-C(34)	119.0(4)
O(9)-C(25)-N(4)	105.1(3)	C(34)-C(33)-H(33)	120.5
O(9)-C(25)-H(25)	107.2	C(33)-C(34)-H(34)	120.8
O(9)-C(25)-C(31)	113.9(3)	C(33)-C(34)-C(35)	118.4(4)
N(4)-C(25)-H(25)	107.2	C(35)-C(34)-H(34)	120.8
N(4)-C(25)-H(25A)	111.8	N(6)-C(35)-C(34)	123.8(4)
C(31)-C(25)-N(4)	115.7(3)	N(6)-C(35)-H(35)	118.1
C(31)-C(25)-H(25)	107.2	C(34)-C(35)-H(35)	118.1
C(31)-C(25)-H(25A)	111.8	O(10)-C(36)-Re(2)	179.2(4)
O(9A)-C(25)-N(4)	96.7(11)	O(11)-C(37)-Re(2)	179.1(3)
O(9A)-C(25)-H(25A)	111.8	O(12)-C(38)-Re(2)	177.1(4)
O(9A)-C(25)-C(31)	108.0(9)	C(1)-O(1)-Re(1)	114.5(2)
N(5)-C(26)-C(23)	118.5(3)	C(4)-O(2)-C(3)	109.1(3)
N(5)-C(26)-C(27)	122.1(4)	C(6)-O(3)-C(5)	107.9(3)
C(27)-C(26)-C(23)	119.2(3)	C(6)-N(1)-Re(1)	119.7(2)
C(26)-C(27)-H(27)	120.6	C(6)-N(1)-C(2)	100.0(3)
C(28)-C(27)-C(26)	118.9(4)	C(2)-N(1)-Re(1)	108.9(2)
C(28)-C(27)-H(27)	120.6	C(4)-N(1)-Re(1)	112.0(2)
C(27)-C(28)-H(28)	120.4	C(4)-N(1)-C(6)	110.8(3)
C(27)-C(28)-C(29)	119.3(4)	C(4)-N(1)-C(2)	103.4(3)
C(29)-C(28)-H(28)	120.4	C(7)-N(2)-Re(1)	117.2(2)
C(28)-C(29)-H(29)	120.7	C(11)-N(2)-Re(1)	124.1(2)
C(30)-C(29)-C(28)	118.7(4)	C(11)-N(2)-C(7)	118.4(3)
C(30)-C(29)-H(29)	120.7	C(16)-N(3)-C(12)	116.6(3)
N(5)-C(30)-C(29)	122.2(4)	N(3)-C(12)-C(6)	114.1(3)
N(5)-C(30)-H(30)	118.9	N(3)-C(12)-C(13)	123.8(3)
C(29)-C(30)-H(30)	118.9	C(13)-C(12)-C(6)	121.8(3)
N(6)-C(31)-C(25)	113.2(3)	C(8)-C(9)-H(9)	120.5
N(6)-C(31)-C(32)	123.8(3)	C(10)-C(9)-H(9)	120.5

C(10)-C(9)-C(8)	118.9(3)	O(1)-C(1)-H(1B)	109.2
N(2)-C(7)-C(8)	122.0(3)	O(1)-C(1)-C(2)	112.1(3)
N(2)-C(7)-C(4)	117.0(3)	H(1A)-C(1)-H(1B)	107.9
C(8)-C(7)-C(4)	120.9(3)	C(2)-C(1)-H(1A)	109.2
C(13)-C(14)-H(14)	120.8	C(2)-C(1)-H(1B)	109.2
C(15)-C(14)-H(14)	120.8	C(14)-C(15)-H(15)	120.6
C(15)-C(14)-C(13)	118.5(4)	C(16)-C(15)-C(14)	118.7(4)
N(2)-C(11)-H(11)	118.8	C(16)-C(15)-H(15)	120.6
N(2)-C(11)-C(10)	122.3(4)	N(1)-C(2)-C(5)	101.6(3)
C(10)-C(11)-H(11)	118.8	N(1)-C(2)-C(1)	109.1(3)
O(4)-C(17)-Re(1)	176.1(3)	N(1)-C(2)-C(3)	103.2(3)
C(9)-C(8)-C(7)	119.1(4)	C(1)-C(2)-C(5)	113.0(3)
C(9)-C(8)-H(8)	120.5	C(1)-C(2)-C(3)	112.4(3)
C(7)-C(8)-H(8)	120.5	C(3)-C(2)-C(5)	116.2(3)
O(6)-C(19)-Re(1)	178.7(3)	O(5)-C(18)-Re(1)	174.9(3)
C(9)-C(10)-C(11)	119.2(3)	O(2)-C(3)-C(2)	106.3(3)
C(9)-C(10)-H(10)	120.4	O(2)-C(3)-H(3A)	110.5
C(11)-C(10)-H(10)	120.4	O(2)-C(3)-H(3B)	110.5
O(3)-C(5)-H(5A)	110.5	C(2)-C(3)-H(3A)	110.5
O(3)-C(5)-H(5B)	110.5	C(2)-C(3)-H(3B)	110.5
O(3)-C(5)-C(2)	106.3(3)	H(3A)-C(3)-H(3B)	108.7
H(5A)-C(5)-H(5B)	108.7	O(2)-C(4)-N(1)	106.7(3)
C(2)-C(5)-H(5A)	110.5	O(2)-C(4)-C(7)	111.6(3)
C(2)-C(5)-H(5B)	110.5	O(2)-C(4)-H(4)	109.0
O(3)-C(6)-N(1)	103.7(3)	N(1)-C(4)-C(7)	111.3(3)
O(3)-C(6)-C(12)	111.8(3)	N(1)-C(4)-H(4)	109.0
O(3)-C(6)-H(6)	108.2	C(7)-C(4)-H(4)	109.0
N(1)-C(6)-C(12)	116.4(3)	N(3)-C(16)-C(15)	123.9(4)
N(1)-C(6)-H(6)	108.2	N(3)-C(16)-H(16)	118.0
C(12)-C(6)-H(6)	108.2	C(15)-C(16)-H(16)	118.0
C(12)-C(13)-C(14)	118.3(3)	C(25)-O(9A)-C(24A)	108.8(15)
C(12)-C(13)-H(13)	120.8	C(21)-C(24A)-H(24C)	111.0
C(14)-C(13)-H(13)	120.8	C(21)-C(24A)-H(24D)	111.0
O(1)-C(1)-H(1A)	109.2	O(9A)-C(24A)-C(21)	103.9(14)

O(9A)-C(24A)-H(24C)	111.0	C(23)-O(8A)-C(22A)	108.3(17)
O(9A)-C(24A)-H(24D)	111.0	C(21)-C(20A)-H(20C)	109.9
H(24C)-C(24A)-H(24D)	109.0	C(21)-C(20A)-H(20D)	109.9
C(21)-C(22A)-H(22C)	110.3	H(20C)-C(20A)-H(20D)	108.3
C(21)-C(22A)-H(22D)	110.3	O(7A)-C(20A)-C(21)	109.1(15)
H(22C)-C(22A)-H(22D)	108.6	O(7A)-C(20A)-H(20C)	109.9
O(8A)-C(22A)-C(21)	107.1(16)	O(7A)-C(20A)-H(20D)	109.9
O(8A)-C(22A)-H(22C)	110.3	C(20A)-O(7A)-Re(2)	113.7(13)
O(8A)-C(22A)-H(22D)	110.3		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **10**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Re1	11(1)	9(1)	13(1)	-2(1)	-1(1)	-1(1)
Re2	13(1)	12(1)	15(1)	-3(1)	0(1)	0(1)
O7	20(2)	17(2)	20(2)	-5(2)	-1(1)	-6(2)
O8	23(2)	17(4)	11(3)	-5(3)	-3(2)	5(2)
O9	35(2)	13(1)	8(3)	0(1)	-9(2)	-6(1)
O10	24(2)	32(2)	24(2)	-6(1)	7(1)	1(1)
O11	23(1)	28(2)	23(1)	-10(1)	-5(1)	-7(1)
O12	35(2)	24(2)	30(2)	-5(1)	-10(1)	14(1)
N4	12(1)	11(1)	15(2)	-2(1)	-1(1)	0(1)
N5	14(1)	10(1)	22(2)	-1(1)	0(1)	-2(1)
N6	21(2)	13(2)	23(2)	-3(1)	-2(1)	-2(1)
C20	15(2)	20(2)	19(2)	-9(2)	-6(2)	-4(2)
C21	19(2)	15(2)	14(2)	-6(1)	-1(1)	-3(1)
C22	17(2)	16(2)	15(2)	-2(2)	-6(2)	-3(2)
C23	15(2)	12(2)	12(2)	-4(1)	1(1)	3(1)
C24	26(3)	18(2)	19(3)	-6(2)	-6(2)	-2(2)
C25	22(2)	13(2)	16(2)	-3(1)	-4(1)	-4(1)
C26	14(2)	13(2)	17(2)	-6(1)	-3(1)	-3(1)
C27	17(2)	16(2)	17(2)	-5(1)	-2(1)	0(1)
C28	22(2)	15(2)	19(2)	-3(2)	-4(2)	-2(1)
C29	26(2)	15(2)	22(2)	1(2)	1(2)	-5(2)
C30	21(2)	18(2)	20(2)	1(2)	6(2)	-1(2)
C31	19(2)	13(2)	14(2)	-2(1)	-1(1)	-6(1)
C32	19(2)	14(2)	25(2)	-1(2)	-4(2)	-2(1)
C33	24(2)	13(2)	26(2)	-6(2)	0(2)	-2(2)
C34	21(2)	18(2)	20(2)	-6(2)	0(2)	-6(2)
C35	18(2)	15(2)	24(2)	-1(2)	-5(2)	-5(1)
C36	18(2)	19(2)	25(2)	-7(2)	-3(2)	-1(2)
C37	19(2)	16(2)	15(2)	-2(1)	2(1)	3(1)
C38	21(2)	22(2)	17(2)	-4(2)	-1(2)	1(2)
O4	25(1)	19(1)	12(1)	-1(1)	-2(1)	-9(1)
O6	27(2)	13(1)	30(2)	-10(1)	-4(1)	0(1)

O5	16(1)	27(2)	27(2)	-3(1)	-7(1)	-2(1)
O1	12(1)	19(1)	15(1)	-3(1)	-3(1)	-2(1)
O2	22(1)	16(1)	13(1)	-1(1)	0(1)	2(1)
O3	15(1)	10(1)	28(2)	-3(1)	-4(1)	1(1)
N1	13(1)	10(1)	10(1)	1(1)	2(1)	-3(1)
N2	16(1)	7(1)	14(1)	-3(1)	-3(1)	1(1)
N3	12(1)	13(2)	23(2)	-1(1)	0(1)	-3(1)
C12	14(2)	14(2)	15(2)	0(1)	-1(1)	-3(1)
C9	20(2)	11(2)	27(2)	-4(2)	-5(2)	2(1)
C7	13(2)	14(2)	17(2)	-8(1)	-2(1)	-1(1)
C14	18(2)	17(2)	25(2)	-5(2)	-2(2)	-7(1)
C11	18(2)	14(2)	16(2)	0(1)	-5(1)	-2(1)
C17	14(2)	9(2)	22(2)	-5(1)	-6(1)	-2(1)
C8	17(2)	14(2)	18(2)	-8(1)	-1(1)	-2(1)
C19	12(2)	18(2)	19(2)	1(2)	-2(1)	0(1)
C10	17(2)	11(2)	22(2)	-1(1)	-3(1)	-4(1)
C5	16(2)	12(2)	22(2)	-3(1)	-3(1)	-1(1)
C6	12(2)	9(2)	18(2)	-1(1)	0(1)	-3(1)
C13	17(2)	15(2)	18(2)	-5(1)	-1(1)	-1(1)
C1	13(2)	17(2)	11(2)	-2(1)	-2(1)	-2(1)
C15	14(2)	22(2)	22(2)	-1(2)	-1(1)	-6(1)
C2	14(2)	11(2)	12(2)	1(1)	-3(1)	0(1)
C18	19(2)	11(2)	18(2)	-1(1)	2(2)	-3(1)
C3	16(2)	18(2)	18(2)	-2(2)	0(1)	1(1)
C4	16(2)	12(2)	12(2)	-3(1)	-1(1)	0(1)
C16	15(2)	16(2)	22(2)	-2(2)	-2(1)	0(1)
O9A	37(4)	14(3)	8(4)	0(3)	-8(4)	-6(3)
C24A	26(4)	17(4)	17(4)	-6(3)	-7(4)	-1(4)
C22A	19(4)	16(4)	17(4)	-2(3)	-4(3)	-2(3)
O8A	22(3)	17(5)	12(4)	-5(4)	-2(3)	3(3)
C20A	16(3)	19(3)	17(3)	-9(3)	-5(3)	-4(3)
O7A	19(3)	16(4)	20(3)	-5(3)	-2(3)	-8(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **11**.

	x	y	z	U(eq)

H20A	9964	4107	2214	20
H20B	10085	2616	2077	20
H22A	7262	4993	2919	19
H22B	7695	5442	2016	19
H23	5354	3508	1547	16
H23A	5335	3526	1565	16
H24A	8806	2266	3267	24
H24B	7202	2726	3416	24
H25	5905	2177	2601	20
H25A	5999	2110	2681	20
H27	4838	6157	1021	20
H28	5263	7449	-163	22
H29	7201	6851	-1018	26
H30	8586	4940	-689	25
H32	7782	-671	2033	23
H33	6822	-2165	1462	25
H34	4751	-1495	955	23
H35	3704	603	1054	23
H9	7435	7949	7033	23
H14	3870	-328	5607	23
H11	8491	6262	5080	19
H8	6769	5972	7668	19
H10	8282	8090	5717	20
H5A	9590	568	6554	20
H5B	9078	195	7454	20
H6	6278	1431	7464	16
H13	5954	-282	6075	20
H1A	9624	3804	6829	16
H1B	10634	2480	6866	16
H15	2190	1469	5713	23
H3A	7925	1699	8252	21
H3B	8873	2848	8108	21
H4	5696	3831	7064	16
H16	2639	3216	6257	21

H24C	9532	2048	2763	24
H24D	8211	2336	3377	24
H22C	6433	3849	3171	20
H22D	7237	5081	2864	20
H20C	8617	5031	1543	19
H20D	9681	4543	2153	19

Table 6. Torsion angles [°] for **10**.

Re1-O1-C1-C2	39.1(3)	N4-C21-C22-O8	26.5(6)
Re1-N1-C6-O3	73.1(3)	N4-C21-C24-O9	20.9(5)
Re1-N1-C6-C12	-50.1(4)	N4-C21-C24A-O9A	-6(2)
Re1-N1-C2-C5	-89.1(3)	N4-C21-C22A-O8A	-29(4)
Re1-N1-C2-C1	30.5(3)	N4-C21-C20A-O7A	-53.1(17)
Re1-N1-C2-C3	150.2(2)	N4-C23-C26-N5	14.2(5)
Re1-N1-C4-O2	-150.4(2)	N4-C23-C26-C27	-169.7(3)
Re1-N1-C4-C7	-28.3(3)	N4-C23-O8A-C22A	-1(6)
Re1-N2-C7-C8	173.3(3)	N4-C25-C31-N6	-77.2(4)
Re1-N2-C7-C4	-9.6(4)	N4-C25-C31-C32	106.2(4)
Re1-N2-C11-C10	-174.5(3)	N4-C25-O9A-C24A	49(2)
Re2-O7-C20-C21	-56.3(4)	N5-C26-C27-C28	-1.7(6)
Re2-N4-C21-C20	-4.4(4)	N6-C31-C32-C33	-0.2(6)
Re2-N4-C21-C22	116.9(3)	C20-C21-C22-O8	144.8(6)
Re2-N4-C21-C24	-122.1(3)	C20-C21-C24-O9	-95.7(4)
Re2-N4-C21-C24A	-96.6(12)	C21-N4-C23-O8	-25.2(8)
Re2-N4-C21-C22A	146.3(10)	C21-N4-C23-C26	97.9(3)
Re2-N4-C21-C20A	31.5(9)	C21-N4-C23-O8A	-18(4)
Re2-N4-C23-O8	-142.3(7)	C21-N4-C25-O9	-36.3(4)
Re2-N4-C23-C26	-19.3(3)	C21-N4-C25-C31	-162.8(3)
Re2-N4-C23-O8A	-135(4)	C21-N4-C25-O9A	-49.1(11)
Re2-N4-C25-O9	84.7(3)	C21-C22A-O8A-C23	20(6)
Re2-N4-C25-C31	-41.8(4)	C21-C20A-O7A-Re2	50(2)
Re2-N4-C25-O9A	71.9(11)	C22-O8-C23-N4	42.7(9)
Re2-N5-C26-C23	-1.0(4)	C22-O8-C23-C26	-80.9(7)
Re2-N5-C26-C27	-177.0(3)	C22-C21-C24-O9	132.7(4)
Re2-N5-C30-C29	177.9(3)	C23-O8-C22-C21	-43.4(9)
O7-C20-C21-N4	38.2(5)	C23-N4-C21-C20	-122.7(3)
O7-C20-C21-C22	-76.5(5)	C23-N4-C21-C22	-1.4(4)
O7-C20-C21-C24	150.6(4)	C23-N4-C21-C24	119.6(3)
O8-C23-C26-N5	133.0(5)	C23-N4-C21-C24A	145.1(12)
O8-C23-C26-C27	-50.8(6)	C23-N4-C21-C22A	28.0(10)
O9-C25-C31-N6	160.9(4)	C23-N4-C21-C20A	-86.7(9)
O9-C25-C31-C32	-15.8(6)	C23-N4-C25-O9	-145.7(3)

C23-N4-C25-C31	87.7(4)	O3-C5-C2-C1	-134.5(3)
C23-N4-C25-O9A	-158.6(10)	O3-C5-C2-C3	93.3(4)
C23-C26-C27-C28	-177.7(3)	N1-C2-C3-O2	-18.4(4)
C24-O9-C25-N4	50.7(4)	N2-C7-C8-C9	1.7(6)
C24-O9-C25-C31	178.4(4)	N2-C7-C4-O2	144.6(3)
C24-C21-C22-O8	-85.0(6)	N2-C7-C4-N1	25.4(4)
C25-O9-C24-C21	-44.4(5)	N2-C11-C10-C9	1.6(6)
C25-N4-C21-C20	125.9(3)	N3-C12-C6-O3	-176.8(3)
C25-N4-C21-C22	-112.8(3)	N3-C12-C6-N1	-57.9(4)
C25-N4-C21-C24	8.2(4)	N3-C12-C13-C14	-1.9(6)
C25-N4-C21-C24A	33.7(12)	C12-N3-C16-C15	3.5(6)
C25-N4-C21-C22A	-83.4(10)	C7-N2-C11-C10	-0.7(5)
C25-N4-C21-C20A	161.9(9)	C14-C15-C16-N3	-3.1(6)
C25-N4-C23-O8	81.5(7)	C11-N2-C7-C8	-0.9(5)
C25-N4-C23-C26	-155.4(3)	C11-N2-C7-C4	176.2(3)
C25-N4-C23-O8A	88(4)	C8-C9-C10-C11	-0.8(6)
C25-C31-C32-C33	176.1(4)	C8-C7-C4-O2	-38.2(5)
C25-O9A-C24A-C21	-28(3)	C8-C7-C4-N1	-157.4(3)
C26-N5-C30-C29	-2.4(6)	C10-C9-C8-C7	-0.7(6)
C26-C23-O8A-C22A	-123(5)	C5-O3-C6-N1	35.5(4)
C26-C27-C28-C29	-0.9(6)	C5-O3-C6-C12	161.6(3)
C27-C28-C29-C30	1.8(6)	C5-C2-C3-O2	-128.6(3)
C28-C29-C30-N5	-0.1(6)	C6-O3-C5-C2	-10.7(4)
C30-N5-C26-C23	179.3(3)	C6-N1-C2-C5	37.2(3)
C30-N5-C26-C27	3.3(5)	C6-N1-C2-C1	156.8(3)
C31-N6-C35-C34	1.5(6)	C6-N1-C2-C3	-83.5(3)
C31-C25-O9A-C24A	168.5(17)	C6-N1-C4-O2	73.2(3)
C31-C32-C33-C34	1.0(6)	C6-N1-C4-C7	-164.8(3)
C32-C33-C34-C35	-0.7(6)	C6-C12-C13-C14	171.6(3)
C33-C34-C35-N6	-0.7(6)	C13-C12-C6-O3	9.1(5)
C35-N6-C31-C25	-177.7(3)	C13-C12-C6-N1	128.0(4)
C35-N6-C31-C32	-1.1(6)	C13-C14-C15-C16	0.1(6)
O1-C1-C2-N1	-46.1(4)	C1-C2-C3-O2	99.0(3)
O1-C1-C2-C5	66.1(4)	C15-C14-C13-C12	2.2(6)
O1-C1-C2-C3	-160.0(3)	C2-N1-C6-O3	-45.6(3)
O3-C5-C2-N1	-17.9(4)	C2-N1-C6-C12	-168.8(3)

C2-N1-C4-O2	-33.2(3)
C2-N1-C4-C7	88.8(3)
C3-O2-C4-N1	22.1(4)
C3-O2-C4-C7	-99.7(3)
C4-O2-C3-C2	-1.8(4)
C4-N1-C6-O3	-154.2(3)
C4-N1-C6-C12	82.6(4)
C4-N1-C2-C5	151.6(3)
C4-N1-C2-C1	-88.8(3)
C4-N1-C2-C3	30.9(3)
C4-C7-C8-C9	-175.4(3)
C16-N3-C12-C6	-174.8(3)
C16-N3-C12-C13	-0.9(6)
O9A-C25-C31-N6	175.8(13)
O9A-C25-C31-C32	-0.9(13)
C24A-C21-C22A-O8A	-138(4)
C24A-C21-C20A-O7A	63(2)
C22A-C21-C24A-O9A	102(2)
C22A-C21-C20A-O7A	-161.2(15)
O8A-C23-C26-N5	133.7(18)
O8A-C23-C26-C27	-50.1(18)
C20A-C21-C24A-O9A	-124.1(18)
C20A-C21-C22A-O8A	83(4)