Electronic Supplementary Material (ESI) for Dalton Transactions.

Cationic Rhenium(III) Complexes: Synthesis, Characterization, and Reactivity for Hydrosilylation of Aldehydes

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Table of Contests:

Table S1. Selected Crystallographic Data and Collection Parameters for **3a** and **4b**

	3a	**4b**	**6**
Empirical Form	$C_{54}H_{29}BF_{34}N_5ORe$	$C_{28}H_{40.50}BF_4N_{5.50}ORe$	$C_{18}H_{12}Cl_2F_{10}N_3ORe$
Formula weight (g/mol)	1691.76	755.18	732.40
crystal system	monoclinic	monoclinic	orthorhombic
space group	P 2_1/c	P1 21/c 1	P b c a
a, Å	9.777(2)	22.3852(5)	12.2754(4)
b, Å	16.855(4)	14.7691(3)	18.8021(6)
c, Å	36.649(8)	19.6678(5)	18.9382(6)
Volume, (Å^3)	6021(2)	6305.8(3)	4371.0(2)
Z	4	8	8
ρ (g/cm^3)	1.866	1.591	2.226
crystal size (mm)	0.39 × 0.13 × 0.09	0.043 × 0.089 × 0.268	0.13 × 0.10 × 0.04
R1, w R2	0.0509, 0.1101	0.0270, 0.0542	0.0323, 0.0609
GOF	1.030	1.009	1.021

Experimental for $C_{54}H_{29}BF_{34}N_5ORe$ (**3a**)

Data Collection and Processing. The sample (**3a**) was submitted by Jessica Smeltz of the Ison research group at North Carolina State University. The sample was mounted on a Mitegen polyimide micromount with a small amount of Paratone N oil. All X-ray measurements were made on a Bruker-Nonius Kappa Axis X8 Apex2 diffractometer at a temperature of 110 K. The unit cell dimensions were determined from a symmetry constrained fit of 9952 reflections with $4.82° < 2\theta < 54.26°$. The data collection strategy was a number of ω and φ scans which collected data up to 63.28° (2θ). The frame integration was performed using SAINT.[4] The resulting raw data was scaled and absorption corrected using a multi-scan averaging of symmetry equivalent data using SADABS.[5]

Structure Solution and Refinement. The structure was solved by direct methods using the XS program.[6] Most non-hydrogen atoms were obtained from the initial solution with the missing atom positions recovered from a subsequent difference Fourier map. The hydrogen atoms were introduced at idealized positions and were allowed to ride on the parent atom. The structural model was fit to the data using full matrix least-squares based on F^2. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation. The structure was refined using the XL program from SHELXTL,[7] graphic plots were produced using the NRCVAX crystallographic program suite. Additional information and other relevant literature references can be found in the reference section of the Facility's Web page (http://www.xray.ncsu.edu).

Acknowledgement
The authors wish to thank the Department of Chemistry of North Carolina State University and the State of North Carolina for funding the purchase of the Apex2 diffractometer.

4 Bruker-Nonius, SAINT version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

5 Bruker-Nonius, SADABS version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

6 Bruker-AXS, XS version 2009.9, **2009**, Bruker-AXS, Madison, WI 53711, USA

7 Bruker-AXS, XL version 2009.9, **2009**, Bruker-AXS, Madison, WI 53711, USA

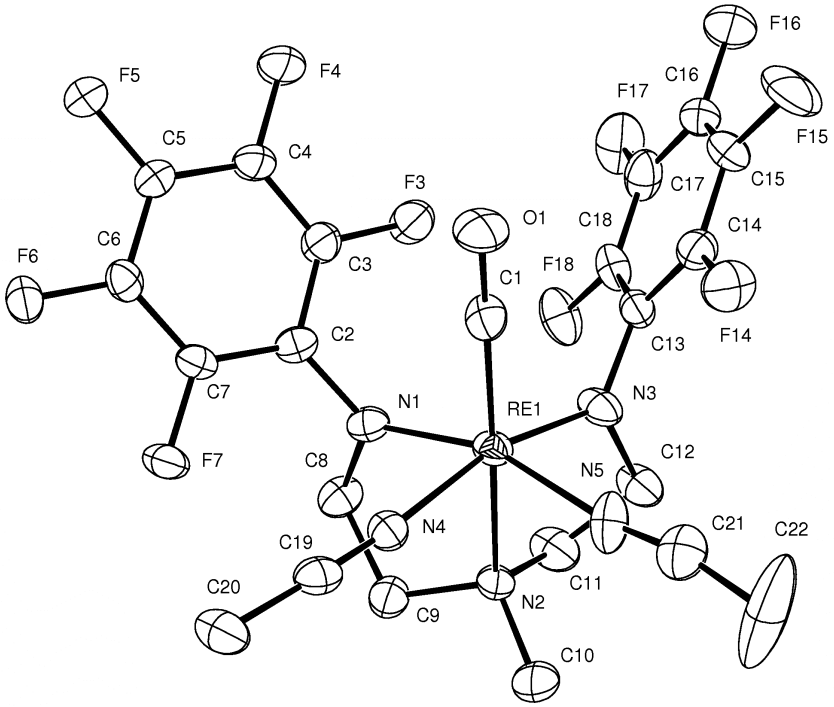


Figure S1. ORTEP drawing of **3a** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

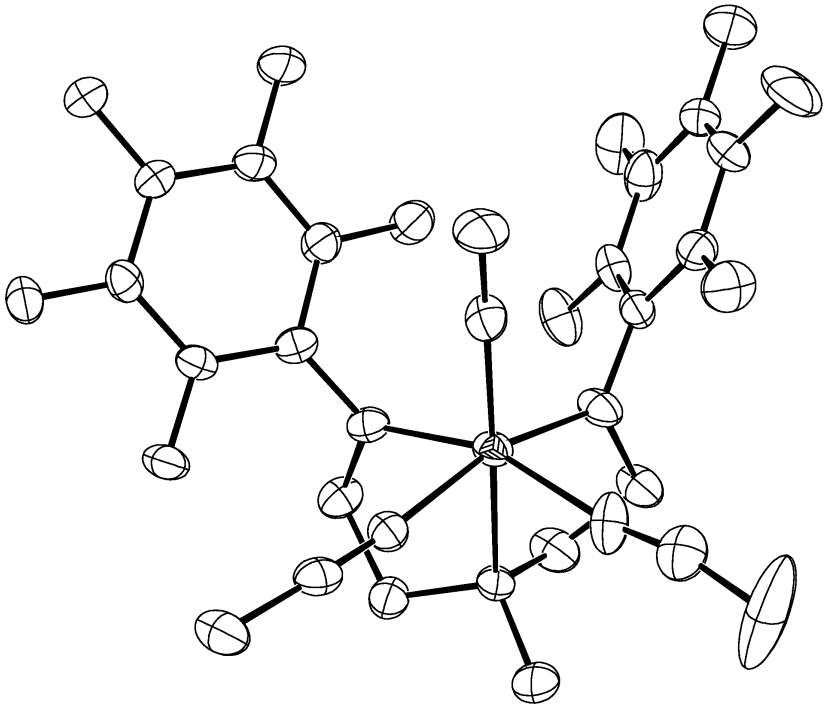


Figure S2. ORTEP drawing of **3a**. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

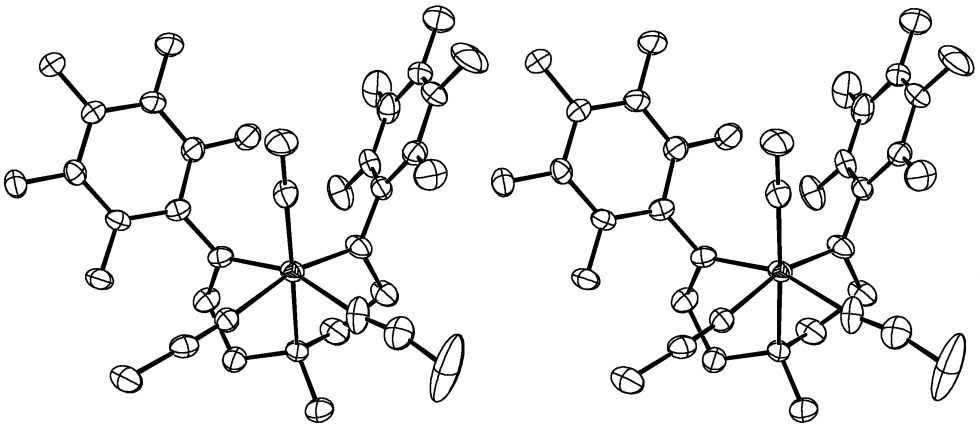


Figure S3. Stereoscopic ORTEP drawing of **3a**. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

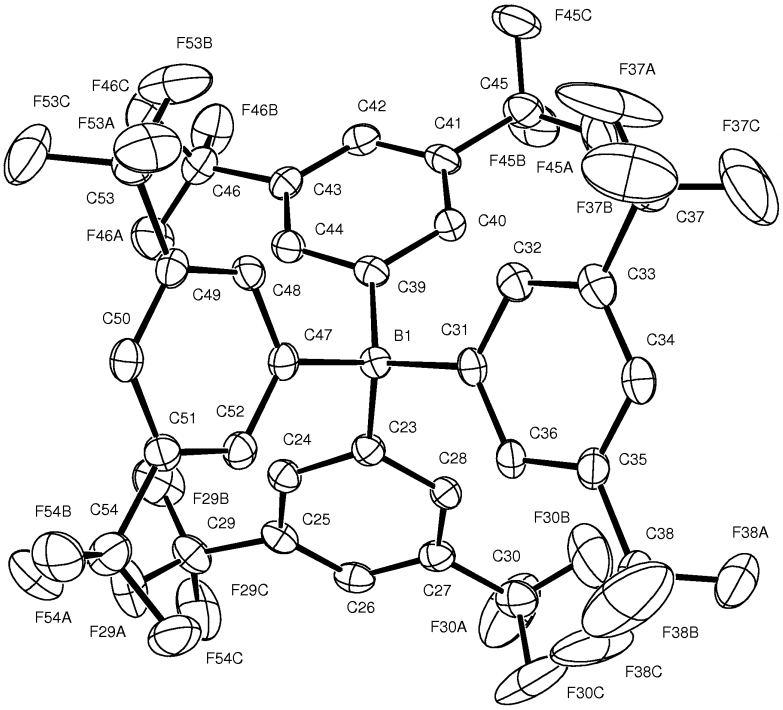


Figure S4. ORTEP drawing of **3a counterion** (BAr^F_4) showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

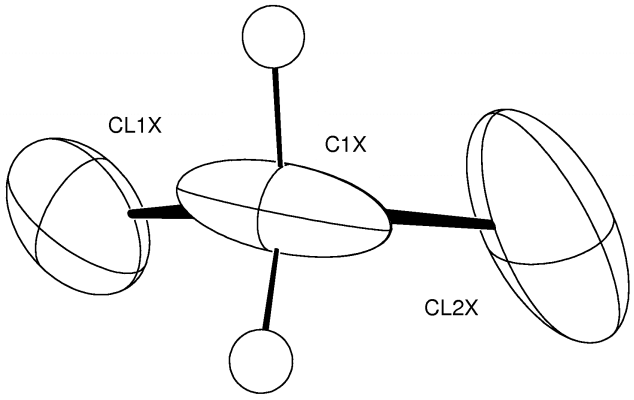


Figure S5. ORTEP drawing of **3a solvent** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table S2. Summary of Crystal Data for **3a**

Formula	$C_{55}H_{31}BCl_2F_{34}N_5ORe$
Formula Weight (*g/mol*)	1691.76
Crystal Dimensions (*mm*)	0.39 × 0.13 × 0.09
Crystal Color and Habit	orange prism
Crystal System	monoclinic
Space Group	P 2_1/c
Temperature, K	110
a, Å	9.777(2)
b, Å	16.855(4)
c, Å	36.649(8)
α,°	90.00
β,°	94.471(9)
γ,°	90.00
V, $Å^3$	6021(2)
Number of reflections to determine final unit cell	9952
Min and Max 2θ for cell determination, °	4.82, 54.26
Z	4
F(000)	3296
ρ (*g/cm*)	1.866
λ, Å, (MoKα)	0.71073
μ, (cm^{-1})	2.259
Diffractometer Type	Bruker-Nonius Kappa Axis X8 Apex2
Scan Type(s)	omega and phi scans
Max 2θ for data collection, °	63.28
Measured fraction of data	0.992
Number of reflections measured	97273
Unique reflections measured	19314
R_{merge}	0.0446
Number of reflections included in refinement	19314
Cut off Threshold Expression	>2sigma(I)
Structure refined using	full matrix least-squares using F^2
Weighting Scheme	calc w=1/[sigma^2(Fo^2)+(0.0524P)^2+8.3576P] where P=(Fo^2+2Fc^2)/3
Number of parameters in least-squares	895
R_1	0.0509
wR_2	0.1101
R_1 (all data)	0.0907
wR_2 (all data)	0.1242
GOF	1.030
Maximum shift/error	0.001
Min & Max peak heights on final ΔF Map (e^-/Å)	-1.657, 1.571

Where:

$R_1 = \Sigma(|F_o| - |F_c|) / \Sigma F_o$

$wR_2 = [\Sigma(w(F_o^2 - F_c^2)^2) / \Sigma(w F_o^4)]^{½}$

$GOF = [\Sigma(w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{½}$

Table S3. Atomic Coordinates for **3a**

Atom	x	y	z	$U_{iso/equiv}$
Re1	0.707117(15)	0.687237(10)	0.121710(4)	0.02466(5)
C1	0.8564(4)	0.7559(2)	0.12944(11)	0.0295(9)
O1	0.9523(3)	0.79695(18)	0.13389(9)	0.0401(8)
N1	0.7563(3)	0.6532(2)	0.07413(9)	0.0265(7)
N2	0.5479(3)	0.5947(2)	0.11019(9)	0.0269(7)
N3	0.7790(3)	0.6064(2)	0.15512(10)	0.0327(8)
N4	0.5846(3)	0.7764(2)	0.09170(9)	0.0251(7)
N5	0.5937(4)	0.7360(2)	0.16330(10)	0.0393(9)
C2	0.8576(4)	0.6887(2)	0.05427(11)	0.0256(8)
C3	0.9970(4)	0.6826(3)	0.06557(12)	0.0314(9)
C4	1.0947(4)	0.7223(3)	0.04817(12)	0.0309(9)
C5	1.0589(4)	0.7692(2)	0.01821(11)	0.0260(8)
C6	0.9230(4)	0.7757(2)	0.00577(10)	0.0258(8)
C7	0.8250(4)	0.7352(2)	0.02372(10)	0.0242(8)
F3	1.0373(2)	0.63852(17)	0.09488(7)	0.0423(7)
F4	1.2288(2)	0.71638(18)	0.06011(8)	0.0467(7)
F5	1.1542(2)	0.80975(14)	0.00153(7)	0.0339(6)
F6	0.8869(2)	0.82250(15)	-0.02269(6)	0.0335(6)
F7	0.6924(2)	0.74475(15)	0.01157(6)	0.0334(6)
C8	0.6741(4)	0.5908(3)	0.05446(12)	0.0325(9)
C9	0.5335(4)	0.5871(3)	0.06931(11)	0.0340(10)
C10	0.4103(4)	0.6109(3)	0.12296(13)	0.0402(11)
C11	0.6049(5)	0.5194(3)	0.12699(14)	0.0418(11)
C12	0.6861(5)	0.5404(3)	0.16263(13)	0.0390(11)
C13	0.9147(4)	0.5987(2)	0.17168(11)	0.0266(8)
C14	0.9716(4)	0.6497(3)	0.19795(11)	0.0308(9)
C15	1.1067(5)	0.6418(3)	0.21233(12)	0.0395(11)
C16	1.1863(4)	0.5808(3)	0.20084(12)	0.0420(12)
C17	1.1330(5)	0.5285(3)	0.17488(12)	0.0401(11)
C18	0.9977(5)	0.5381(3)	0.16064(11)	0.0333(10)
F14	0.8975(3)	0.70986(16)	0.21007(7)	0.0426(7)
F15	1.1577(3)	0.6920(2)	0.23813(9)	0.0629(9)
F16	1.3161(3)	0.5737(2)	0.21510(8)	0.0645(10)
F17	1.2100(3)	0.46943(19)	0.16365(8)	0.0573(8)
F18	0.9497(3)	0.48692(16)	0.13512(7)	0.0461(7)
C19	0.5239(4)	0.8166(3)	0.07155(12)	0.0285(9)
C20	0.4507(4)	0.8691(3)	0.04534(13)	0.0358(10)
C21	0.5479(5)	0.7624(3)	0.18803(13)	0.0389(11)
C22	0.4672(10)	0.7964(4)	0.21509(19)	0.104(3)
B1	0.9522(4)	0.2167(3)	0.13141(12)	0.0219(8)
C23	0.9383(4)	0.2788(2)	0.09730(10)	0.0227(8)
C24	0.8138(4)	0.2894(2)	0.07628(10)	0.0239(8)
C25	0.7937(4)	0.3483(3)	0.04977(11)	0.0274(8)
C26	0.8990(4)	0.3984(2)	0.04202(11)	0.0292(9)
C27	1.0247(4)	0.3891(2)	0.06230(11)	0.0266(8)

C28	1.0429(4)	0.3321(2)	0.08954(10)	0.0234(8)
C29	0.6539(5)	0.3571(3)	0.03028(12)	0.0342(10)
F29A	0.6105(3)	0.29250(17)	0.01262(9)	0.0519(8)
F29B	0.5589(3)	0.3750(2)	0.05314(8)	0.0676(10)
F29C	0.6475(3)	0.4160(2)	0.00586(9)	0.0685(10)
C30	1.1414(5)	0.4408(3)	0.05423(12)	0.0360(10)
F30A	1.1035(4)	0.5148(2)	0.04534(13)	0.0937(14)
F30B	1.2347(4)	0.4497(3)	0.08099(10)	0.0985(16)
F30C	1.2028(4)	0.4185(2)	0.02603(12)	0.114(2)
C31	1.1093(4)	0.1846(2)	0.14142(10)	0.0230(7)
C32	1.1545(4)	0.1557(2)	0.17585(11)	0.0267(8)
C33	1.2828(4)	0.1204(2)	0.18271(11)	0.0291(9)
C34	1.3703(4)	0.1130(3)	0.15537(11)	0.0291(9)
C35	1.3279(4)	0.1398(2)	0.12061(11)	0.0263(8)
C36	1.1992(4)	0.1747(2)	0.11387(11)	0.0249(8)
C37	1.3259(5)	0.0891(3)	0.22010(13)	0.0424(12)
F37A	1.2478(5)	0.1056(3)	0.24492(8)	0.127(2)
F37B	1.3348(5)	0.0105(2)	0.22022(9)	0.0853(12)
F37C	1.4529(4)	0.1092(3)	0.23106(11)	0.1080(17)
C38	1.4206(4)	0.1303(3)	0.09036(13)	0.0382(11)
F38A	1.5448(3)	0.1568(2)	0.09875(9)	0.0647(9)
F38B	1.4439(5)	0.0540(3)	0.08355(13)	0.1107(18)
F38C	1.3760(4)	0.1598(4)	0.06002(9)	0.143(3)
C39	0.8859(4)	0.2653(2)	0.16443(10)	0.0221(8)
C40	0.9643(4)	0.3072(2)	0.19177(10)	0.0242(8)
C41	0.9037(4)	0.3539(2)	0.21723(10)	0.0269(8)
C42	0.7624(4)	0.3629(2)	0.21566(11)	0.0269(8)
C43	0.6833(4)	0.3239(2)	0.18861(11)	0.0241(8)
C44	0.7437(4)	0.2745(2)	0.16396(10)	0.0252(8)
C45	0.9896(5)	0.3951(3)	0.24722(12)	0.0348(10)
F45A	1.1222(3)	0.3821(2)	0.24544(8)	0.0655(10)
F45B	0.9721(3)	0.47377(18)	0.24625(8)	0.0554(8)
F45C	0.9570(3)	0.37283(18)	0.28023(7)	0.0516(8)
C46	0.5301(4)	0.3334(3)	0.18522(12)	0.0316(9)
F46A	0.4784(3)	0.33795(18)	0.15045(8)	0.0471(7)
F46B	0.4898(3)	0.39861(16)	0.20210(8)	0.0495(7)
F46C	0.4662(3)	0.27240(17)	0.20002(8)	0.0469(7)
C47	0.8679(3)	0.1338(2)	0.12195(10)	0.0219(8)
C48	0.8172(4)	0.0881(2)	0.14934(10)	0.0221(8)
C49	0.7601(4)	0.0140(2)	0.14269(11)	0.0230(8)
C50	0.7510(4)	-0.0177(2)	0.10753(11)	0.0235(8)
C51	0.7991(4)	0.0268(2)	0.07953(10)	0.0240(8)
C52	0.8568(4)	0.1012(2)	0.08667(11)	0.0238(8)
C53	0.7075(4)	-0.0345(3)	0.17291(11)	0.0293(9)
F53A	0.7745(3)	-0.10176(16)	0.17884(7)	0.0466(7)
F53B	0.7105(4)	0.00323(17)	0.20480(8)	0.0604(9)
F53C	0.5756(3)	-0.0559(2)	0.16514(9)	0.0603(9)
C54	0.7812(4)	-0.0028(3)	0.04115(12)	0.0319(9)
F54A	0.6646(3)	0.02410(17)	0.02328(7)	0.0476(7)
F54B	0.7719(3)	-0.08225(15)	0.03925(7)	0.0401(6)

F54C	0.8818(3)	0.01877(17)	0.02059(7)	0.0501(8)
C1X	0.1462(6)	0.9431(4)	0.1212(2)	0.079(2)
Cl1X	0.12453(16)	0.94941(12)	0.07401(6)	0.0870(6)
Cl2X	0.2811(2)	0.8762(2)	0.13455(6)	0.1392(11)
H8A	0.7208	0.5390	0.0579	0.039
H8B	0.6644	0.6030	0.0280	0.039
H9A	0.4751	0.6305	0.0586	0.041
H9B	0.4888	0.5360	0.0624	0.041
H10A	0.3468	0.5687	0.1144	0.060
H10B	0.3766	0.6620	0.1132	0.060
H10C	0.4167	0.6127	0.1498	0.060
H11A	0.6652	0.4932	0.1102	0.050
H11B	0.5294	0.4826	0.1317	0.050
H12A	0.6232	0.5566	0.1812	0.047
H12B	0.7397	0.4939	0.1721	0.047
H20A	0.4648	0.9243	0.0532	0.054
H20B	0.3525	0.8566	0.0440	0.054
H20C	0.4854	0.8618	0.0212	0.054
H22A	0.5276	0.8239	0.2336	0.156
H22B	0.4174	0.7542	0.2268	0.156
H22C	0.4015	0.8342	0.2034	0.156
H24	0.7396	0.2549	0.0803	0.029
H26	0.8865	0.4378	0.0235	0.035
H28	1.1289	0.3289	0.1034	0.028
H32	1.0961	0.1602	0.1953	0.032
H34	1.4585	0.0898	0.1602	0.035
H36	1.1715	0.1922	0.0898	0.030
H40	1.0615	0.3035	0.1929	0.029
H42	0.7211	0.3953	0.2329	0.032
H44	0.6864	0.2462	0.1463	0.030
H48	0.8221	0.1084	0.1736	0.027
H50	0.7126	-0.0688	0.1029	0.028
H52	0.8896	0.1307	0.0671	0.029
H1X1	0.0600	0.9246	0.1309	0.095
H1X2	0.1681	0.9963	0.1316	0.095

Table S4. Anisotropic Displacement Parameters for **3a**

Atom	u^{11}	u^{22}	u^{33}	u^{12}	u^{13}	u^{23}
Re1	0.02228(7)	0.02352(9)	0.02752(8)	-0.00443(7)	-0.00228(5)	0.00084(7)
C1	0.038(2)	0.021(2)	0.029(2)	-0.0052(18)	0.0007(17)	0.0026(17)
O1	0.0384(17)	0.0329(18)	0.048(2)	-0.0154(14)	-0.0024(15)	-0.0026(15)
N1	0.0226(15)	0.0228(17)	0.0341(19)	-0.0021(14)	0.0031(14)	0.0006(15)
N2	0.0243(16)	0.0301(19)	0.0258(17)	-0.0083(14)	-0.0012(13)	-0.0011(14)
N3	0.0310(18)	0.0276(19)	0.037(2)	-0.0098(15)	-0.0095(15)	0.0070(16)
N4	0.0255(16)	0.0241(18)	0.0257(17)	-0.0010(14)	0.0013(13)	0.0004(14)
N5	0.060(3)	0.036(2)	0.0226(18)	0.0004(19)	0.0092(18)	-0.0015(16)
C2	0.0230(17)	0.0219(19)	0.032(2)	0.0006(16)	0.0054(15)	-0.0022(17)
C3	0.0274(19)	0.033(2)	0.034(2)	0.0050(18)	0.0035(17)	0.0107(19)
C4	0.0197(18)	0.037(2)	0.036(2)	0.0029(17)	0.0020(16)	0.005(2)
C5	0.0243(18)	0.027(2)	0.028(2)	-0.0017(16)	0.0065(15)	-0.0003(17)
C6	0.0290(19)	0.029(2)	0.0193(18)	0.0028(17)	0.0013(15)	-0.0040(16)
C7	0.0198(17)	0.027(2)	0.0252(19)	0.0019(15)	-0.0009(14)	-0.0066(16)
F3	0.0290(13)	0.0509(17)	0.0474(16)	0.0096(12)	0.0060(11)	0.0224(14)
F4	0.0198(12)	0.070(2)	0.0497(17)	0.0038(12)	0.0002(11)	0.0196(15)
F5	0.0270(12)	0.0430(15)	0.0323(13)	-0.0061(11)	0.0059(10)	0.0040(11)
F6	0.0342(13)	0.0425(16)	0.0236(12)	0.0035(11)	0.0010(10)	0.0072(11)
F7	0.0205(11)	0.0456(16)	0.0331(13)	-0.0001(10)	-0.0040(9)	-0.0023(12)
C8	0.034(2)	0.027(2)	0.038(2)	-0.0048(18)	0.0078(18)	-0.0047(19)
C9	0.035(2)	0.038(3)	0.029(2)	-0.0121(19)	0.0024(18)	-0.0033(19)
C10	0.031(2)	0.050(3)	0.040(3)	-0.016(2)	0.0027(19)	0.000(2)
C11	0.042(2)	0.030(3)	0.052(3)	-0.011(2)	-0.009(2)	0.012(2)
C12	0.040(2)	0.031(2)	0.044(3)	-0.014(2)	-0.012(2)	0.015(2)
C13	0.0288(19)	0.028(2)	0.0226(19)	-0.0038(17)	-0.0023(16)	0.0021(16)
C14	0.031(2)	0.033(2)	0.029(2)	-0.0055(18)	0.0045(17)	-0.0043(18)
C15	0.034(2)	0.054(3)	0.029(2)	-0.012(2)	-0.0058(18)	-0.008(2)
C16	0.023(2)	0.071(4)	0.031(2)	0.000(2)	0.0005(17)	0.003(2)
C17	0.043(3)	0.047(3)	0.031(2)	0.017(2)	0.009(2)	0.006(2)
C18	0.044(2)	0.032(2)	0.022(2)	0.000(2)	-0.0040(18)	-0.0008(17)
F14	0.0427(15)	0.0406(15)	0.0455(16)	-0.0008(12)	0.0093(12)	-0.0167(13)
F15	0.0512(18)	0.082(3)	0.0518(19)	-0.0153(16)	-0.0170(15)	-0.0274(17)
F16	0.0264(14)	0.112(3)	0.0543(19)	0.0043(16)	-0.0050(12)	0.0139(19)
F17	0.0635(19)	0.057(2)	0.0528(18)	0.0307(16)	0.0133(15)	0.0063(15)
F18	0.0710(19)	0.0331(15)	0.0323(14)	0.0037(14)	-0.0081(13)	-0.0093(12)
C19	0.0228(18)	0.029(2)	0.034(2)	-0.0056(17)	0.0018(16)	-0.0046(19)
C20	0.029(2)	0.031(2)	0.045(3)	-0.0015(18)	-0.0081(19)	0.005(2)
C21	0.047(3)	0.030(2)	0.040(3)	-0.007(2)	0.006(2)	0.001(2)
C22	0.171(8)	0.088(5)	0.064(4)	0.054(5)	0.079(5)	0.023(4)
B1	0.0197(18)	0.026(2)	0.020(2)	-0.0001(17)	0.0012(15)	-0.0004(17)
C23	0.0247(17)	0.022(2)	0.0220(18)	-0.0006(15)	0.0027(15)	-0.0030(15)
C24	0.0266(18)	0.0213(19)	0.0235(19)	-0.0009(15)	0.0007(15)	-0.0016(15)
C25	0.034(2)	0.026(2)	0.0221(19)	0.0025(18)	-0.0007(16)	-0.0038(16)
C26	0.042(2)	0.025(2)	0.0214(19)	0.0006(18)	0.0042(17)	0.0023(16)
C27	0.033(2)	0.024(2)	0.0242(19)	-0.0032(17)	0.0060(16)	-0.0041(16)
C28	0.0251(18)	0.023(2)	0.0223(18)	-0.0041(15)	0.0015(15)	-0.0060(15)

C29	0.039(2)	0.033(3)	0.029(2)	0.005(2)	-0.0077(19)	0.0007(19)
F29A	0.0384(15)	0.0432(17)	0.070(2)	0.0041(13)	-0.0196(14)	-0.0177(15)
F29B	0.0392(16)	0.110(3)	0.0526(19)	0.0341(17)	-0.0050(14)	-0.0127(19)
F29C	0.070(2)	0.060(2)	0.069(2)	-0.0093(17)	-0.0367(17)	0.0305(18)
C30	0.047(3)	0.028(2)	0.033(2)	-0.007(2)	0.006(2)	-0.0060(19)
F30A	0.079(3)	0.040(2)	0.167(4)	-0.0115(18)	0.042(3)	0.032(2)
F30B	0.090(3)	0.125(4)	0.075(2)	-0.081(3)	-0.030(2)	0.050(2)
F30C	0.135(4)	0.100(3)	0.121(3)	-0.086(3)	0.106(3)	-0.078(3)
C31	0.0211(16)	0.0218(19)	0.0255(19)	-0.0052(15)	-0.0023(14)	-0.0039(16)
C32	0.0263(19)	0.027(2)	0.026(2)	-0.0018(17)	-0.0011(16)	-0.0012(17)
C33	0.0291(19)	0.026(2)	0.031(2)	-0.0007(17)	-0.0052(17)	0.0015(17)
C34	0.0203(17)	0.031(2)	0.035(2)	0.0026(16)	-0.0049(16)	0.0009(18)
C35	0.0187(17)	0.030(2)	0.030(2)	-0.0023(16)	-0.0005(15)	-0.0021(17)
C36	0.0192(16)	0.029(2)	0.026(2)	-0.0048(15)	-0.0025(15)	-0.0007(16)
C37	0.042(3)	0.046(3)	0.037(3)	0.012(2)	-0.009(2)	0.010(2)
F37A	0.144(4)	0.213(5)	0.0257(17)	0.135(4)	0.017(2)	0.030(2)
F37B	0.143(4)	0.057(2)	0.056(2)	0.019(2)	0.007(2)	0.0273(18)
F37C	0.093(3)	0.138(4)	0.083(3)	-0.041(3)	-0.056(2)	0.063(3)
C38	0.0193(19)	0.058(3)	0.037(3)	0.0061(19)	-0.0002(17)	-0.004(2)
F38A	0.0316(15)	0.102(3)	0.062(2)	-0.0201(16)	0.0153(14)	-0.0211(19)
F38B	0.116(3)	0.084(3)	0.145(4)	-0.032(3)	0.089(3)	-0.063(3)
F38C	0.055(2)	0.335(8)	0.041(2)	0.097(3)	0.0275(17)	0.060(3)
C39	0.0291(18)	0.0176(18)	0.0195(17)	-0.0008(15)	0.0007(15)	0.0015(14)
C40	0.0231(17)	0.027(2)	0.0226(18)	-0.0027(16)	0.0001(14)	0.0010(16)
C41	0.036(2)	0.026(2)	0.0176(18)	-0.0043(18)	-0.0011(16)	-0.0002(16)
C42	0.034(2)	0.022(2)	0.026(2)	-0.0013(17)	0.0085(17)	-0.0026(16)
C43	0.0241(17)	0.021(2)	0.028(2)	0.0005(15)	0.0048(15)	0.0031(15)
C44	0.0294(19)	0.023(2)	0.0228(19)	-0.0049(16)	0.0013(15)	-0.0005(16)
C45	0.042(2)	0.036(3)	0.027(2)	-0.005(2)	0.0053(19)	-0.0085(19)
F45A	0.0393(16)	0.107(3)	0.0482(18)	-0.0083(17)	-0.0071(13)	-0.0392(19)
F45B	0.085(2)	0.0358(17)	0.0444(17)	-0.0198(16)	-0.0004(15)	-0.0107(13)
F45C	0.072(2)	0.062(2)	0.0203(13)	-0.0195(16)	-0.0026(13)	-0.0007(13)
C46	0.029(2)	0.028(2)	0.038(2)	-0.0027(17)	0.0087(18)	-0.0016(18)
F46A	0.0338(14)	0.0637(19)	0.0428(16)	0.0114(13)	-0.0025(12)	0.0004(14)
F46B	0.0372(14)	0.0391(17)	0.074(2)	0.0059(12)	0.0156(14)	-0.0196(15)
F46C	0.0360(14)	0.0421(17)	0.0639(19)	-0.0094(12)	0.0116(13)	0.0076(14)
C47	0.0156(15)	0.023(2)	0.0265(19)	0.0019(14)	0.0001(14)	-0.0011(16)
C48	0.0192(16)	0.026(2)	0.0207(18)	0.0014(15)	-0.0004(14)	-0.0009(15)
C49	0.0175(16)	0.024(2)	0.028(2)	-0.0009(15)	0.0004(14)	0.0035(16)
C50	0.0208(17)	0.0181(19)	0.031(2)	-0.0024(15)	0.0003(15)	-0.0019(16)
C51	0.0217(17)	0.026(2)	0.0242(19)	0.0017(15)	0.0022(15)	-0.0046(16)
C52	0.0193(16)	0.026(2)	0.026(2)	0.0013(15)	0.0018(14)	-0.0014(16)
C53	0.031(2)	0.027(2)	0.029(2)	-0.0005(17)	0.0013(17)	0.0018(17)
F53A	0.0550(17)	0.0420(16)	0.0432(16)	0.0188(14)	0.0072(13)	0.0197(13)
F53B	0.109(3)	0.0381(17)	0.0393(16)	-0.0151(17)	0.0365(17)	-0.0057(13)
F53C	0.0324(14)	0.073(2)	0.074(2)	-0.0171(14)	-0.0029(13)	0.0422(18)
C54	0.033(2)	0.028(2)	0.036(2)	-0.0035(18)	0.0100(18)	-0.0043(19)
F54A	0.0551(17)	0.0522(18)	0.0328(14)	0.0084(14)	-0.0127(13)	-0.0063(13)
F54B	0.0535(16)	0.0283(14)	0.0387(15)	-0.0039(12)	0.0051(12)	-0.0113(11)
F54C	0.0582(18)	0.0548(19)	0.0405(15)	-0.0213(15)	0.0245(13)	-0.0184(14)

C1X	0.053(4)	0.060(4)	0.126(6)	-0.030(3)	0.023(4)	-0.043(4)
Cl1X	0.0472(8)	0.0976(14)	0.1158(16)	0.0201(9)	0.0029(9)	-0.0302(12)
Cl2X	0.0496(10)	0.280(4)	0.0865(15)	0.0051(15)	-0.0063(9)	0.0034(19)

Table S5. Bond Lengths for **3a**

Re1-C1	1.867(4)	C25-C29	1.500(6)
Re1-N3	1.928(3)	C26-C27	1.394(6)
Re1-N1	1.932(3)	C26-H26	0.9500
Re1-N5	2.120(4)	C27-C28	1.387(5)
Re1-N4	2.167(3)	C27-C30	1.484(6)
Re1-N2	2.220(3)	C28-H28	0.9500
C1-O1	1.166(5)	C29-F29A	1.319(5)
N1-C2	1.408(5)	C29-F29B	1.333(5)
N1-C8	1.476(5)	C29-F29C	1.335(5)
N2-C10	1.484(5)	C30-F30C	1.292(5)
N2-C11	1.499(6)	C30-F30B	1.295(6)
N2-C9	1.499(5)	C30-F30A	1.334(6)
N3-C13	1.421(5)	C31-C32	1.392(5)
N3-C12	1.475(5)	C31-C36	1.399(5)
N4-C19	1.136(5)	C32-C33	1.393(5)
N5-C21	1.133(5)	C32-H32	0.9500
C2-C7	1.384(6)	C33-C34	1.373(6)
C2-C3	1.397(6)	C33-C37	1.498(6)
C3-F3	1.340(5)	C34-C35	1.384(6)
C3-C4	1.364(6)	C34-H34	0.9500
C4-F4	1.354(4)	C35-C36	1.394(5)
C4-C5	1.375(6)	C35-C38	1.494(6)
C5-F5	1.341(4)	C36-H36	0.9500
C5-C6	1.375(5)	C37-F37A	1.263(6)
C6-F6	1.333(4)	C37-F37C	1.320(6)
C6-C7	1.384(5)	C37-F37B	1.327(6)
C7-F7	1.348(4)	C38-F38C	1.264(6)
C8-C9	1.519(6)	C38-F38A	1.309(5)
C8-H8A	0.9900	C38-F38B	1.333(6)
C8-H8B	0.9900	C39-C44	1.398(5)
C9-H9A	0.9900	C39-C40	1.404(5)
C9-H9B	0.9900	C40-C41	1.388(5)
C10-H10A	0.9800	C40-H40	0.9500
C10-H10B	0.9800	C41-C42	1.386(6)
C10-H10C	0.9800	C41-C45	1.501(6)
C11-C12	1.516(6)	C42-C43	1.376(6)
C11-H11A	0.9900	C42-H42	0.9500
C11-H11B	0.9900	C43-C44	1.393(5)
C12-H12A	0.9900	C43-C46	1.502(6)
C12-H12B	0.9900	C44-H44	0.9500
C13-C14	1.376(6)	C45-F45A	1.322(5)
C13-C18	1.384(6)	C45-F45C	1.329(5)
C14-F14	1.342(5)	C45-F45B	1.336(5)
C14-C15	1.390(6)	C46-F46B	1.336(5)
C15-F15	1.336(5)	C46-F46A	1.336(5)
C15-C16	1.375(7)	C46-F46C	1.339(5)
C16-F16	1.339(5)	C47-C48	1.387(5)
C16-C17	1.370(7)	C47-C52	1.401(5)

C17-F17	1.333(5)	C48-C49	1.381(5)
C17-C18	1.393(6)	C48-H48	0.9500
C18-F18	1.331(5)	C49-C50	1.392(5)
C19-C20	1.452(6)	C49-C53	1.500(5)
C20-H20A	0.9800	C50-C51	1.383(5)
C20-H20B	0.9800	C50-H50	0.9500
C20-H20C	0.9800	C51-C52	1.391(5)
C21-C22	1.433(7)	C51-C54	1.490(6)
C22-H22A	0.9800	C52-H52	0.9500
C22-H22B	0.9800	C53-F53A	1.319(5)
C22-H22C	0.9800	C53-F53B	1.329(5)
B1-C23	1.628(6)	C53-F53C	1.348(5)
B1-C39	1.637(6)	C54-F54C	1.335(5)
B1-C31	1.642(6)	C54-F54B	1.343(5)
B1-C47	1.645(6)	C54-F54A	1.349(5)
C23-C24	1.400(5)	C1X-Cl1X	1.730(8)
C23-C28	1.407(5)	C1X-Cl2X	1.775(8)
C24-C25	1.392(6)	C1X-H1X1	0.9900
C24-H24	0.9500	C1X-H1X2	0.9900
C25-C26	1.378(6)		

Table S6. Bond Angles for **3a**

C1-Re1-N3	95.74(16)	C23-C24-H24	118.5
C1-Re1-N1	94.26(16)	C26-C25-C24	120.9(4)
N3-Re1-N1	105.04(15)	C26-C25-C29	120.6(4)
C1-Re1-N5	95.58(17)	C24-C25-C29	118.5(4)
N3-Re1-N5	90.15(16)	C25-C26-C27	117.6(4)
N1-Re1-N5	160.96(15)	C25-C26-H26	121.2
C1-Re1-N4	92.40(15)	C27-C26-H26	121.2
N3-Re1-N4	166.44(14)	C28-C27-C26	121.3(4)
N1-Re1-N4	85.09(13)	C28-C27-C30	119.6(4)
N5-Re1-N4	78.26(14)	C26-C27-C30	119.1(4)
C1-Re1-N2	172.91(16)	C27-C28-C23	122.2(4)
N3-Re1-N2	80.77(13)	C27-C28-H28	118.9
N1-Re1-N2	80.79(13)	C23-C28-H28	118.9
N5-Re1-N2	90.62(14)	F29A-C29-F29B	106.4(4)
N4-Re1-N2	92.22(13)	F29A-C29-F29C	106.8(4)
O1-C1-Re1	177.9(4)	F29B-C29-F29C	104.7(4)
C2-N1-C8	115.3(3)	F29A-C29-C25	113.5(4)
C2-N1-Re1	125.1(3)	F29B-C29-C25	112.1(3)
C8-N1-Re1	119.2(2)	F29C-C29-C25	112.7(4)
C10-N2-C11	110.1(3)	F30C-C30-F30B	107.2(5)
C10-N2-C9	108.3(3)	F30C-C30-F30A	102.4(4)
C11-N2-C9	109.9(3)	F30B-C30-F30A	104.0(4)
C10-N2-Re1	116.8(3)	F30C-C30-C27	113.6(4)
C11-N2-Re1	106.5(2)	F30B-C30-C27	115.1(4)
C9-N2-Re1	105.1(2)	F30A-C30-C27	113.3(4)
C13-N3-C12	114.8(3)	C32-C31-C36	116.0(3)
C13-N3-Re1	128.1(3)	C32-C31-B1	122.9(3)
C12-N3-Re1	116.9(3)	C36-C31-B1	120.5(3)
C19-N4-Re1	169.9(3)	C31-C32-C33	122.1(4)
C21-N5-Re1	171.6(4)	C31-C32-H32	119.0
C7-C2-C3	116.1(3)	C33-C32-H32	119.0
C7-C2-N1	122.1(3)	C34-C33-C32	120.7(4)
C3-C2-N1	121.7(4)	C34-C33-C37	119.2(4)
F3-C3-C4	118.3(4)	C32-C33-C37	120.0(4)
F3-C3-C2	119.8(4)	C33-C34-C35	118.9(4)
C4-C3-C2	121.8(4)	C33-C34-H34	120.6
F4-C4-C3	120.6(4)	C35-C34-H34	120.6
F4-C4-C5	118.7(4)	C34-C35-C36	120.1(4)
C3-C4-C5	120.8(4)	C34-C35-C38	119.5(4)
F5-C5-C4	121.0(3)	C36-C35-C38	120.3(4)
F5-C5-C6	119.7(4)	C35-C36-C31	122.1(4)
C4-C5-C6	119.3(4)	C35-C36-H36	118.9
F6-C6-C5	119.8(4)	C31-C36-H36	118.9
F6-C6-C7	120.8(3)	F37A-C37-F37C	109.4(5)
C5-C6-C7	119.3(4)	F37A-C37-F37B	105.1(5)
F7-C7-C2	119.4(3)	F37C-C37-F37B	101.2(4)
F7-C7-C6	117.9(3)	F37A-C37-C33	116.0(4)
C2-C7-C6	122.7(3)	F37C-C37-C33	112.2(4)

N1-C8-C9	109.2(3)	F37B-C37-C33	111.6(4)
N1-C8-H8A	109.8	F38C-C38-F38A	108.8(5)
C9-C8-H8A	109.8	F38C-C38-F38B	105.5(5)
N1-C8-H8B	109.8	F38A-C38-F38B	101.8(4)
C9-C8-H8B	109.8	F38C-C38-C35	115.1(4)
H8A-C8-H8B	108.3	F38A-C38-C35	113.2(4)
N2-C9-C8	109.7(3)	F38B-C38-C35	111.4(4)
N2-C9-H9A	109.7	C44-C39-C40	116.1(3)
C8-C9-H9A	109.7	C44-C39-B1	119.8(3)
N2-C9-H9B	109.7	C40-C39-B1	123.7(3)
C8-C9-H9B	109.7	C41-C40-C39	121.8(4)
H9A-C9-H9B	108.2	C41-C40-H40	119.1
N2-C10-H10A	109.5	C39-C40-H40	119.1
N2-C10-H10B	109.5	C42-C41-C40	120.7(4)
H10A-C10-H10B	109.5	C42-C41-C45	118.6(4)
N2-C10-H10C	109.5	C40-C41-C45	120.7(4)
H10A-C10-H10C	109.5	C43-C42-C41	118.7(4)
H10B-C10-H10C	109.5	C43-C42-H42	120.6
N2-C11-C12	108.0(4)	C41-C42-H42	120.6
N2-C11-H11A	110.1	C42-C43-C44	120.7(4)
C12-C11-H11A	110.1	C42-C43-C46	120.7(4)
N2-C11-H11B	110.1	C44-C43-C46	118.7(4)
C12-C11-H11B	110.1	C43-C44-C39	122.0(4)
H11A-C11-H11B	108.4	C43-C44-H44	119.0
N3-C12-C11	107.7(4)	C39-C44-H44	119.0
N3-C12-H12A	110.2	F45A-C45-F45C	107.7(4)
C11-C12-H12A	110.2	F45A-C45-F45B	106.7(4)
N3-C12-H12B	110.2	F45C-C45-F45B	105.4(4)
C11-C12-H12B	110.2	F45A-C45-C41	112.5(4)
H12A-C12-H12B	108.5	F45C-C45-C41	112.1(4)
C14-C13-C18	116.8(4)	F45B-C45-C41	112.1(4)
C14-C13-N3	123.7(4)	F46B-C46-F46A	106.9(4)
C18-C13-N3	119.5(4)	F46B-C46-F46C	106.3(3)
F14-C14-C13	120.3(4)	F46A-C46-F46C	106.1(4)
F14-C14-C15	117.9(4)	F46B-C46-C43	112.2(4)
C13-C14-C15	121.8(4)	F46A-C46-C43	112.7(3)
F15-C15-C16	120.1(4)	F46C-C46-C43	112.3(4)
F15-C15-C14	119.9(4)	C48-C47-C52	116.3(4)
C16-C15-C14	120.0(4)	C48-C47-B1	121.4(3)
F16-C16-C17	120.7(5)	C52-C47-B1	122.0(3)
F16-C16-C15	119.3(4)	C49-C48-C47	122.5(4)
C17-C16-C15	120.0(4)	C49-C48-H48	118.8
F17-C17-C16	120.1(4)	C47-C48-H48	118.8
F17-C17-C18	121.0(5)	C48-C49-C50	120.4(3)
C16-C17-C18	119.0(4)	C48-C49-C53	121.3(4)
F18-C18-C13	120.0(4)	C50-C49-C53	118.3(3)
F18-C18-C17	117.4(4)	C51-C50-C49	118.6(4)
C13-C18-C17	122.5(4)	C51-C50-H50	120.7
N4-C19-C20	178.0(5)	C49-C50-H50	120.7
C19-C20-H20A	109.5	C50-C51-C52	120.3(4)

C19-C20-H20B	109.5	C50-C51-C54	119.8(4)
H20A-C20-H20B	109.5	C52-C51-C54	119.7(4)
C19-C20-H20C	109.5	C51-C52-C47	121.9(4)
H20A-C20-H20C	109.5	C51-C52-H52	119.0
H20B-C20-H20C	109.5	C47-C52-H52	119.0
N5-C21-C22	169.6(6)	F53A-C53-F53B	106.8(3)
C21-C22-H22A	109.5	F53A-C53-F53C	105.0(3)
C21-C22-H22B	109.5	F53B-C53-F53C	105.6(4)
H22A-C22-H22B	109.5	F53A-C53-C49	113.3(3)
C21-C22-H22C	109.5	F53B-C53-C49	113.8(3)
H22A-C22-H22C	109.5	F53C-C53-C49	111.6(3)
H22B-C22-H22C	109.5	F54C-C54-F54B	107.0(3)
C23-B1-C39	103.4(3)	F54C-C54-F54A	105.4(4)
C23-B1-C31	113.8(3)	F54B-C54-F54A	105.0(3)
C39-B1-C31	114.6(3)	F54C-C54-C51	113.8(3)
C23-B1-C47	111.9(3)	F54B-C54-C51	112.7(4)
C39-B1-C47	111.1(3)	F54A-C54-C51	112.2(3)
C31-B1-C47	102.3(3)	Cl1X-C1X-Cl2X	110.1(4)
C24-C23-C28	114.9(4)	Cl1X-C1X-H1X1	109.6
C24-C23-B1	121.5(3)	Cl2X-C1X-H1X1	109.6
C28-C23-B1	123.2(3)	Cl1X-C1X-H1X2	109.6
C25-C24-C23	123.0(4)	Cl2X-C1X-H1X2	109.6
C25-C24-H24	118.5	H1X1-C1X-H1X2	108.1

Table S7. Torsion Angles for **3a**

N3-Re1-C1-O1	60(11)	C39-B1-C23-C24	-79.2(4)
N1-Re1-C1-O1	-46(11)	C31-B1-C23-C24	155.8(3)
N5-Re1-C1-O1	150(11)	C47-B1-C23-C24	40.5(5)
N4-Re1-C1-O1	-131(11)	C39-B1-C23-C28	92.8(4)
N2-Re1-C1-O1	0(12)	C31-B1-C23-C28	-32.2(5)
C1-Re1-N1-C2	-10.8(3)	C47-B1-C23-C28	-147.6(3)
N3-Re1-N1-C2	-107.9(3)	C28-C23-C24-C25	-0.3(6)
N5-Re1-N1-C2	110.2(5)	B1-C23-C24-C25	172.3(4)
N4-Re1-N1-C2	81.2(3)	C23-C24-C25-C26	2.1(6)
N2-Re1-N1-C2	174.3(3)	C23-C24-C25-C29	-176.8(4)
C1-Re1-N1-C8	176.0(3)	C24-C25-C26-C27	-1.5(6)
N3-Re1-N1-C8	78.9(3)	C29-C25-C26-C27	177.3(4)
N5-Re1-N1-C8	-63.0(6)	C25-C26-C27-C28	-0.6(6)
N4-Re1-N1-C8	-91.9(3)	C25-C26-C27-C30	178.8(4)
N2-Re1-N1-C8	1.1(3)	C26-C27-C28-C23	2.5(6)
C1-Re1-N2-C10	170.6(11)	C30-C27-C28-C23	-176.9(4)
N3-Re1-N2-C10	109.6(3)	C24-C23-C28-C27	-1.9(5)
N1-Re1-N2-C10	-143.3(3)	B1-C23-C28-C27	-174.4(4)
N5-Re1-N2-C10	19.6(3)	C26-C25-C29-F29A	122.0(4)
N4-Re1-N2-C10	-58.7(3)	C24-C25-C29-F29A	-59.2(5)
C1-Re1-N2-C11	47.2(13)	C26-C25-C29-F29B	-117.4(5)
N3-Re1-N2-C11	-13.7(3)	C24-C25-C29-F29B	61.4(5)
N1-Re1-N2-C11	93.3(3)	C26-C25-C29-F29C	0.5(6)
N5-Re1-N2-C11	-103.8(3)	C24-C25-C29-F29C	179.3(4)
N4-Re1-N2-C11	177.9(3)	C28-C27-C30-F30C	99.7(5)
C1-Re1-N2-C9	-69.4(13)	C26-C27-C30-F30C	-79.7(6)
N3-Re1-N2-C9	-130.4(3)	C28-C27-C30-F30B	-24.4(6)
N1-Re1-N2-C9	-23.3(3)	C26-C27-C30-F30B	156.2(4)
N5-Re1-N2-C9	139.6(3)	C28-C27-C30-F30A	-144.0(4)
N4-Re1-N2-C9	61.3(3)	C26-C27-C30-F30A	36.6(6)
C1-Re1-N3-C13	-12.9(4)	C23-B1-C31-C32	156.3(4)
N1-Re1-N3-C13	83.1(4)	C39-B1-C31-C32	37.5(5)
N5-Re1-N3-C13	-108.6(4)	C47-B1-C31-C32	-82.8(4)
N4-Re1-N3-C13	-139.6(5)	C23-B1-C31-C36	-32.9(5)
N2-Re1-N3-C13	160.8(4)	C39-B1-C31-C36	-151.7(3)
C1-Re1-N3-C12	172.2(3)	C47-B1-C31-C36	88.0(4)
N1-Re1-N3-C12	-91.8(3)	C36-C31-C32-C33	1.3(6)
N5-Re1-N3-C12	76.6(3)	B1-C31-C32-C33	172.5(4)
N4-Re1-N3-C12	45.6(8)	C31-C32-C33-C34	0.1(6)
N2-Re1-N3-C12	-14.0(3)	C31-C32-C33-C37	-179.0(4)
C1-Re1-N4-C19	106.4(19)	C32-C33-C34-C35	-1.2(6)
N3-Re1-N4-C19	-126.7(18)	C37-C33-C34-C35	177.9(4)
N1-Re1-N4-C19	12.3(19)	C33-C34-C35-C36	0.9(6)
N5-Re1-N4-C19	-158.4(19)	C33-C34-C35-C38	-178.5(4)
N2-Re1-N4-C19	-68.2(19)	C34-C35-C36-C31	0.6(6)
C1-Re1-N5-C21	-42(3)	C38-C35-C36-C31	180.0(4)
N3-Re1-N5-C21	54(3)	C32-C31-C36-C35	-1.6(6)
N1-Re1-N5-C21	-163(2)	B1-C31-C36-C35	-173.1(4)

N4-Re1-N5-C21	-133(3)	C34-C33-C37-F37A	172.4(5)
N2-Re1-N5-C21	135(3)	C32-C33-C37-F37A	-8.5(7)
C8-N1-C2-C7	66.8(5)	C34-C33-C37-F37C	45.6(6)
Re1-N1-C2-C7	-106.6(4)	C32-C33-C37-F37C	-135.2(5)
C8-N1-C2-C3	-117.0(4)	C34-C33-C37-F37B	-67.3(6)
Re1-N1-C2-C3	69.6(5)	C32-C33-C37-F37B	111.9(5)
C7-C2-C3-F3	-179.8(4)	C34-C35-C38-F38C	-176.9(5)
N1-C2-C3-F3	3.7(6)	C36-C35-C38-F38C	3.7(7)
C7-C2-C3-C4	2.0(6)	C34-C35-C38-F38A	-50.8(6)
N1-C2-C3-C4	-174.4(4)	C36-C35-C38-F38A	129.8(4)
F3-C3-C4-F4	0.7(7)	C34-C35-C38-F38B	63.2(6)
C2-C3-C4-F4	178.9(4)	C36-C35-C38-F38B	-116.2(5)
F3-C3-C4-C5	-179.2(4)	C23-B1-C39-C44	74.9(4)
C2-C3-C4-C5	-1.1(7)	C31-B1-C39-C44	-160.6(3)
F4-C4-C5-F5	-1.5(6)	C47-B1-C39-C44	-45.3(5)
C3-C4-C5-F5	178.4(4)	C23-B1-C39-C40	-97.4(4)
F4-C4-C5-C6	180.0(4)	C31-B1-C39-C40	27.1(5)
C3-C4-C5-C6	-0.1(7)	C47-B1-C39-C40	142.4(4)
F5-C5-C6-F6	-0.2(6)	C44-C39-C40-C41	1.1(6)
C4-C5-C6-F6	178.4(4)	B1-C39-C40-C41	173.7(4)
F5-C5-C6-C7	-178.4(3)	C39-C40-C41-C42	-2.2(6)
C4-C5-C6-C7	0.2(6)	C39-C40-C41-C45	177.2(4)
C3-C2-C7-F7	-179.0(4)	C40-C41-C42-C43	0.7(6)
N1-C2-C7-F7	-2.6(6)	C45-C41-C42-C43	-178.7(4)
C3-C2-C7-C6	-1.9(6)	C41-C42-C43-C44	1.9(6)
N1-C2-C7-C6	174.5(4)	C41-C42-C43-C46	-178.5(4)
F6-C6-C7-F7	-0.2(5)	C42-C43-C44-C39	-3.1(6)
C5-C6-C7-F7	178.0(3)	C46-C43-C44-C39	177.3(4)
F6-C6-C7-C2	-177.3(4)	C40-C39-C44-C43	1.5(6)
C5-C6-C7-C2	0.9(6)	B1-C39-C44-C43	-171.4(4)
C2-N1-C8-C9	-152.3(4)	C42-C41-C45-F45A	-179.6(4)
Re1-N1-C8-C9	21.5(5)	C40-C41-C45-F45A	1.0(6)
C10-N2-C9-C8	166.1(4)	C42-C41-C45-F45C	58.9(5)
C11-N2-C9-C8	-73.6(4)	C40-C41-C45-F45C	-120.5(4)
Re1-N2-C9-C8	40.6(4)	C42-C41-C45-F45B	-59.4(5)
N1-C8-C9-N2	-41.6(5)	C40-C41-C45-F45B	121.2(4)
C10-N2-C11-C12	-90.3(4)	C42-C43-C46-F46B	19.8(5)
C9-N2-C11-C12	150.5(4)	C44-C43-C46-F46B	-160.6(4)
Re1-N2-C11-C12	37.1(4)	C42-C43-C46-F46A	140.5(4)
C13-N3-C12-C11	-136.5(4)	C44-C43-C46-F46A	-39.9(5)
Re1-N3-C12-C11	39.1(5)	C42-C43-C46-F46C	-99.8(5)
N2-C11-C12-N3	-49.0(5)	C44-C43-C46-F46C	79.8(5)
C12-N3-C13-C14	-115.9(5)	C23-B1-C47-C48	-152.9(3)
Re1-N3-C13-C14	69.2(5)	C39-B1-C47-C48	-37.9(5)
C12-N3-C13-C18	65.8(5)	C31-B1-C47-C48	84.9(4)
Re1-N3-C13-C18	-109.2(4)	C23-B1-C47-C52	34.4(5)
C18-C13-C14-F14	-180.0(4)	C39-B1-C47-C52	149.4(3)
N3-C13-C14-F14	1.7(6)	C31-B1-C47-C52	-87.8(4)
C18-C13-C14-C15	1.1(6)	C52-C47-C48-C49	0.7(5)
N3-C13-C14-C15	-177.3(4)	B1-C47-C48-C49	-172.4(3)

F14-C14-C15-F15	1.8(7)	C47-C48-C49-C50	-0.1(6)
C13-C14-C15-F15	-179.2(4)	C47-C48-C49-C53	179.3(4)
F14-C14-C15-C16	179.7(4)	C48-C49-C50-C51	-0.7(5)
C13-C14-C15-C16	-1.3(7)	C53-C49-C50-C51	179.9(4)
F15-C15-C16-F16	-1.7(7)	C49-C50-C51-C52	0.8(6)
C14-C15-C16-F16	-179.6(4)	C49-C50-C51-C54	-175.4(3)
F15-C15-C16-C17	178.7(4)	C50-C51-C52-C47	-0.3(6)
C14-C15-C16-C17	0.9(7)	C54-C51-C52-C47	175.9(3)
F16-C16-C17-F17	0.4(7)	C48-C47-C52-C51	-0.5(5)
C15-C16-C17-F17	179.9(4)	B1-C47-C52-C51	172.6(3)
F16-C16-C17-C18	-179.8(4)	C48-C49-C53-F53A	-116.7(4)
C15-C16-C17-C18	-0.3(7)	C50-C49-C53-F53A	62.7(5)
C14-C13-C18-F18	-179.5(4)	C48-C49-C53-F53B	5.6(5)
N3-C13-C18-F18	-1.1(6)	C50-C49-C53-F53B	-174.9(3)
C14-C13-C18-C17	-0.4(6)	C48-C49-C53-F53C	125.0(4)
N3-C13-C18-C17	178.0(4)	C50-C49-C53-F53C	-55.5(5)
F17-C17-C18-F18	-1.0(6)	C50-C51-C54-F54C	-148.1(4)
C16-C17-C18-F18	179.1(4)	C52-C51-C54-F54C	35.7(5)
F17-C17-C18-C13	179.9(4)	C50-C51-C54-F54B	-26.0(5)
C16-C17-C18-C13	0.0(7)	C52-C51-C54-F54B	157.8(3)
Re1-N4-C19-C20	-73(15)	C50-C51-C54-F54A	92.2(5)
Re1-N5-C21-C22	175(2)	C52-C51-C54-F54A	-84.0(4)

Experimental for $C_{28}H_{40.50}BF_4N_{5.50}ORe$ (**4b**)

An orange-brown rod-like specimen of $C_{29}H_{40.50}BF_4N_{5.50}ORe$, approximate dimensions 0.043 mm x 0.089 mm x 0.268 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 10.10 hours. The frames were integrated with the Bruker SAINT[8] software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 96744 reflections to a maximum θ angle of 30.52° (0.70 Å resolution), of which 19226 were independent (average redundancy 5.032, completeness = 99.9%, R_{int} = 5.17%, R_{sig} = 4.26%) and 15346 (79.82%) were greater than $2\sigma(F^2)$. The final cell constants of a = 22.3852(5) Å, b = 14.7691(3) Å, c = 19.6678(5) Å, β = 104.1230(10)°, volume = 6305.8(3) Å^3, are based upon the refinement of the XYZ-centroids of 1239 reflections above 20 σ(I) with 4.187° < 2θ < 62.04°. Data were corrected for absorption effects using the numerical method (SADABS[9]). The ratio of minimum to maximum apparent transmission was 0.554. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.4210 and 0.8500.

The final anisotropic full-matrix least-squares refinement on F^2 with 812 variables converged at R1 = 2.70%, for the observed data and wR2 = 5.42% for all data. The goodness-of-fit was 1.009. The largest peak in the final difference electron density synthesis was 1.713 e^-/Å^3 and the largest hole was -1.037 e^-/Å^3 with an RMS deviation of 0.127 e^-/Å^3. On the basis of the final model, the calculated density was 1.591 g/cm^3 and F(000), 3016 e^-.

[8] Bruker-Nonius, SAINT version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

[9] Bruker-Nonius, SADABS version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

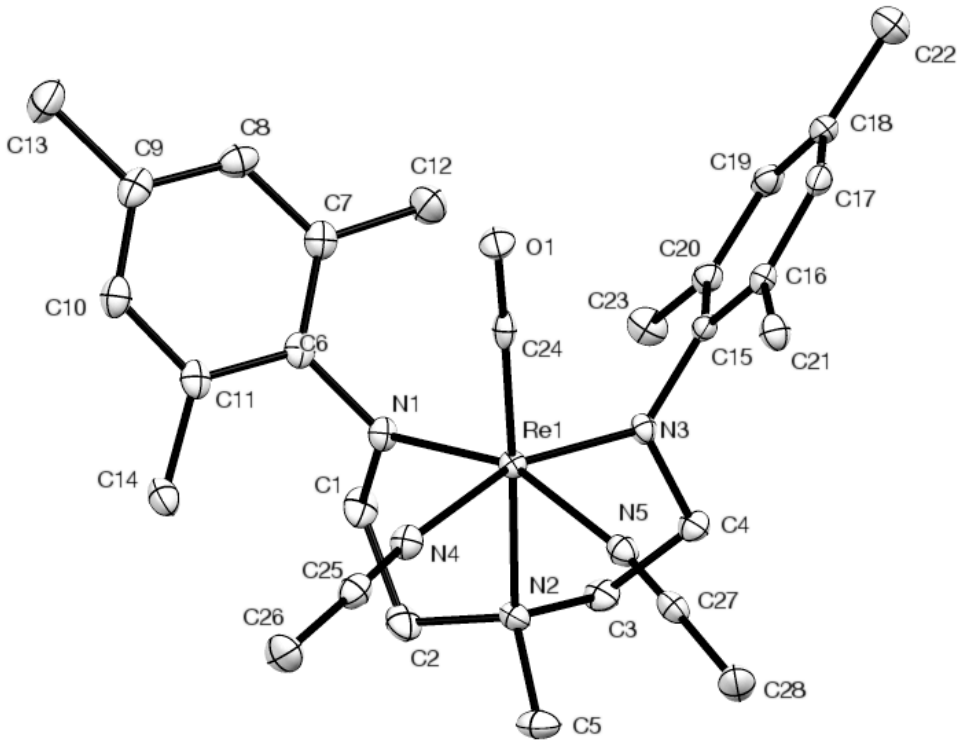


Figure S6. ORTEP drawing of **4b** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

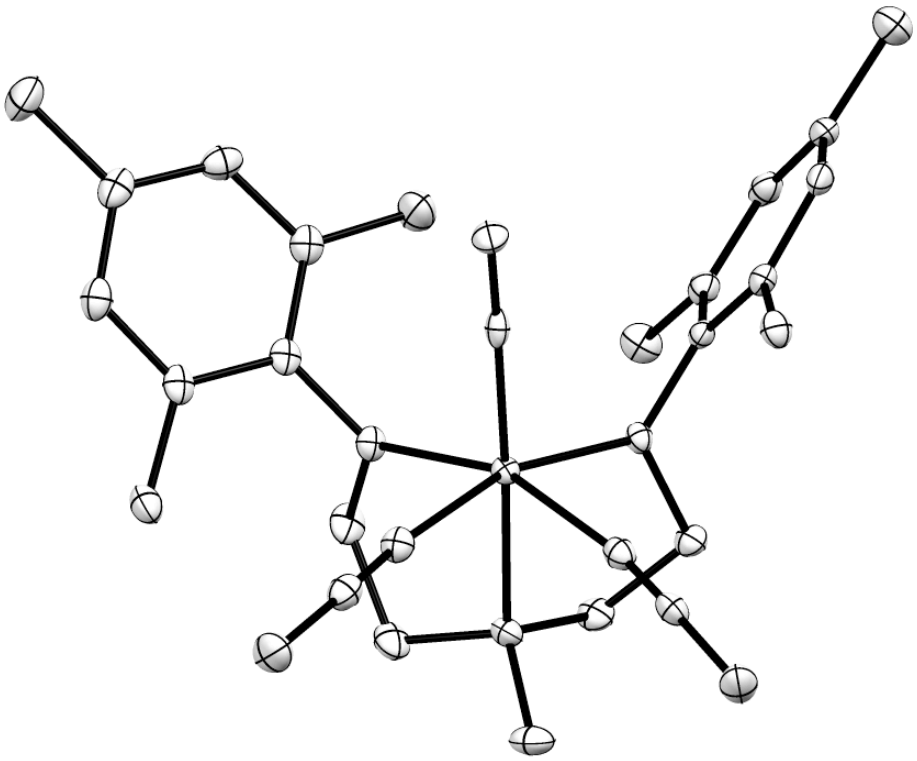


Figure S7. ORTEP drawing of **4b**. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

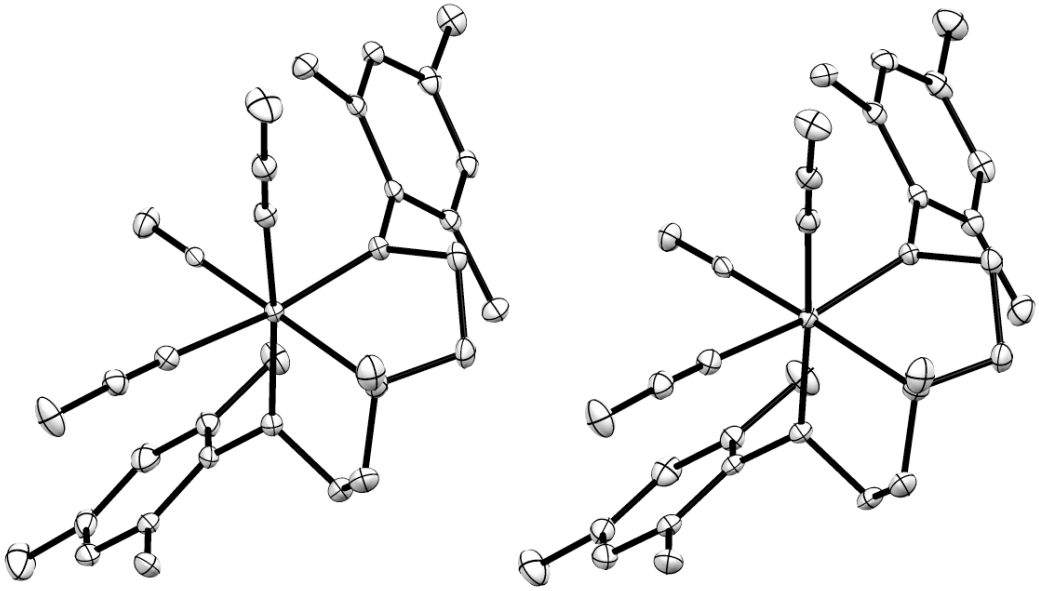


Figure S8. Stereoscopic ORTEP drawing of **3a**. Ellipsoids are at the 50% probability level and hydrogen atoms were omitted for clarity.

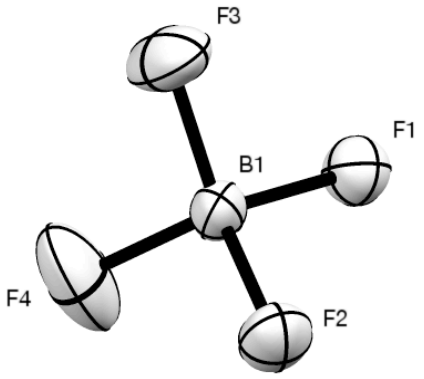


Figure S9. ORTEP drawing of **4b counterion** (BF_4) showing naming and numbering scheme. Ellipsoids are at the 50% probability level.

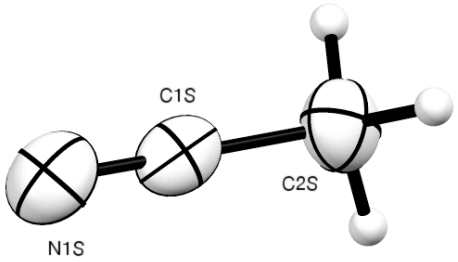


Figure S10. ORTEP drawing of **4b solvent** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table S8. Summary of Crystal Data for **4b**

Chemical formula	$C_{29}H_{40.50}BF_4N_{5.50}ORe$
Formula weight (*g/mol*)	755.18
Temperature (*K*)	100(2)
Wavelength (*Å*)	0.71073
Crystal size (*mm*)	0.043 x 0.089 x 0.268
Crystal habit	orange-brown rod
Crystal system	monoclinic
Space group	P 1 21/c 1
a, Å	22.3852(5)
b, Å	14.7691(3)
c, Å	19.6678(5)
α, °	90
β, °	104.1230(10)
γ, °	90
Volume, $Å^3$	6305.8(3)
Z	8
Density (calculated), g/cm^3	1.591
Absorption coefficient, mm^{-1}	3.909
F(000)	3016
Theta range for data collection	1.85 to 30.52°
Index ranges	-26<=h<=31, -21<=k<=21, -28<=l<=28
Reflections collected	96744
Independent reflections	19226 [R(int) = 0.0517]
Coverage of independent reflections	99.9%
Absorption correction	numerical
Max. and min. transmission	0.8500 and 0.4210
Refinement method	Full-matrix least-squares on F2
Refinement program	SHELXL-2013 (Sheldrick, 2013)
Function minimized	Σ w(Fo2 - Fc2)2
Data / restraints / parameters	19226 / 70 / 812
Goodness-of-fit on F2	1.009
Δ/σmax	0.003
Final R indices	15346 data; I>2σ(I) R1 = 0.0270, wR2 = 0.0495
	all data R1 = 0.0450, wR2 = 0.0542
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0210P)^2+2.3269P]$ where $P=(F_o^2+2F_c^2)/3$
Largest diff. peak and hole, $eÅ^{-3}$	1.713 and -1.037
R.M.S. deviation from mean, $eÅ^{-3}$	0.127

Table S9. Atomic Coordinates and equivalent isotropic atomic displacement parameters ($Å^3$) for **4b**

	x	y	z	U(eq)
Re1	0.28477(2)	0.30308(2)	0.44508(2)	0.01078(2)
O1	0.21319(9)	0.47352(12)	0.39162(10)	0.0187(4)
N1	0.23586(10)	0.27994(13)	0.51176(11)	0.0135(4)
N2	0.32599(10)	0.16980(14)	0.48699(11)	0.0146(4)
N3	0.25238(9)	0.23050(13)	0.36192(11)	0.0118(4)
N4	0.34786(10)	0.37659(14)	0.52530(12)	0.0161(5)
N5	0.36482(10)	0.32421(14)	0.40477(11)	0.0153(4)
C1	0.24160(13)	0.19013(17)	0.54578(15)	0.0190(6)
C2	0.30613(13)	0.15422(18)	0.55280(14)	0.0203(6)
C3	0.29757(12)	0.10101(17)	0.43259(14)	0.0176(5)
C4	0.28560(12)	0.14346(16)	0.36042(14)	0.0160(5)
C5	0.39419(12)	0.16123(18)	0.50237(16)	0.0221(6)
C6	0.19501(12)	0.34041(16)	0.53598(13)	0.0140(5)
C7	0.13591(12)	0.35775(17)	0.49397(14)	0.0171(5)
C8	0.09723(12)	0.41604(18)	0.51906(15)	0.0199(6)
C9	0.11501(13)	0.45662(17)	0.58460(15)	0.0194(6)
C10	0.17328(13)	0.43808(17)	0.62522(14)	0.0187(6)
C11	0.21385(12)	0.38032(16)	0.60280(14)	0.0161(5)
C12	0.11155(14)	0.3127(2)	0.42373(15)	0.0267(7)
C13	0.07125(14)	0.5169(2)	0.61132(17)	0.0285(7)
C14	0.27499(13)	0.36055(18)	0.65295(14)	0.0201(6)
C15	0.20501(11)	0.24741(16)	0.29934(13)	0.0120(5)
C16	0.21128(12)	0.31528(16)	0.25146(13)	0.0139(5)
C17	0.16331(12)	0.32961(17)	0.19235(14)	0.0169(5)
C18	0.11047(12)	0.27665(18)	0.17723(14)	0.0183(5)
C19	0.10599(12)	0.20885(17)	0.22433(14)	0.0176(5)
C20	0.15144(12)	0.19333(17)	0.28510(14)	0.0154(5)
C21	0.26920(12)	0.36924(17)	0.25795(14)	0.0171(5)
C22	0.06104(14)	0.2915(2)	0.11064(15)	0.0259(6)
C23	0.14192(12)	0.11858(17)	0.33388(15)	0.0200(6)
C24	0.24206(12)	0.40880(16)	0.41174(13)	0.0136(5)
C25	0.38466(12)	0.41042(17)	0.56764(15)	0.0181(5)
C26	0.43039(13)	0.4524(2)	0.62402(15)	0.0252(6)
C27	0.41128(12)	0.33091(17)	0.39092(14)	0.0163(5)
C28	0.47120(12)	0.3360(2)	0.37465(15)	0.0228(6)
Re2	0.25015(2)	0.82822(2)	0.55452(2)	0.01163(3)
O2	0.17444(9)	0.99150(12)	0.49104(10)	0.0207(4)
N6	0.19850(10)	0.80079(14)	0.61762(11)	0.0144(4)

	x	y	z	U(eq)
N7	0.29571(10)	0.69904(14)	0.60099(12)	0.0171(5)
N8	0.22383(10)	0.75066(13)	0.47305(11)	0.0137(4)
N9	0.30772(10)	0.90912(14)	0.63619(12)	0.0164(5)
N10	0.33156(10)	0.85702(14)	0.51726(11)	0.0159(4)
C29	0.20653(13)	0.71156(17)	0.65225(14)	0.0194(6)
C30	0.27334(14)	0.68291(18)	0.66522(15)	0.0218(6)
C31	0.27278(13)	0.62623(16)	0.54742(14)	0.0181(6)
C32	0.26087(12)	0.66693(16)	0.47487(14)	0.0167(5)
C33	0.36412(13)	0.69906(18)	0.62003(16)	0.0234(6)
C34	0.15402(12)	0.85751(16)	0.63949(14)	0.0150(5)
C35	0.09670(12)	0.87666(17)	0.59395(15)	0.0177(5)
C36	0.05521(13)	0.93173(18)	0.61744(15)	0.0215(6)
C37	0.06854(14)	0.96672(18)	0.68486(16)	0.0239(6)
C38	0.12527(14)	0.94709(17)	0.72887(15)	0.0215(6)
C39	0.16846(13)	0.89238(17)	0.70814(14)	0.0180(6)
C40	0.07588(13)	0.83581(19)	0.52159(15)	0.0218(6)
C41	0.02245(15)	0.0241(2)	0.71059(18)	0.0341(8)
C42	0.22763(14)	0.87180(18)	0.76129(14)	0.0230(6)
C43	0.17675(12)	0.76118(16)	0.40942(13)	0.0127(5)
C44	0.18209(12)	0.82461(16)	0.35812(13)	0.0137(5)
C45	0.13460(12)	0.83221(17)	0.29767(14)	0.0153(5)
C46	0.08315(12)	0.77569(17)	0.28441(14)	0.0156(5)
C47	0.07997(12)	0.71223(16)	0.33514(14)	0.0158(5)
C48	0.12494(12)	0.70412(16)	0.39756(13)	0.0142(5)
C49	0.23966(12)	0.87958(18)	0.36369(15)	0.0196(6)
C50	0.03508(12)	0.78044(18)	0.21635(14)	0.0206(6)
C51	0.11728(12)	0.63238(17)	0.44948(14)	0.0187(6)
C52	0.20430(12)	0.92934(16)	0.51560(13)	0.0136(5)
C53	0.34151(12)	0.94623(17)	0.67942(14)	0.0167(5)
C54	0.38466(13)	0.9924(2)	0.73641(16)	0.0261(7)
C55	0.37599(13)	0.86755(18)	0.49972(14)	0.0186(6)
C56	0.43206(13)	0.8804(2)	0.47600(17)	0.0289(7)
B1	0.57354(15)	0.3820(2)	0.56462(18)	0.0242(7)
F1	0.51638(8)	0.33748(11)	0.54600(9)	0.0303(4)
F2	0.59178(8)	0.40477(13)	0.50422(9)	0.0338(4)
F3	0.56807(9)	0.45946(14)	0.60201(10)	0.0442(5)
F4	0.61635(9)	0.32377(15)	0.60514(12)	0.0548(6)
B2	0.4894(5)	0.4521(7)	0.8414(4)	0.022(2)
F5	0.4687(5)	0.4933(9)	0.7781(4)	0.044(2)
F6	0.4456(5)	0.4342(8)	0.8763(6)	0.055(2)

	x	y	z	U(eq)
F7	0.5327(6)	0.5088(8)	0.8833(5)	0.052(2)
F8	0.5202(6)	0.3724(7)	0.8347(9)	0.054(2)
B2A	0.4791(11)	0.4526(11)	0.8347(10)	0.053(4)
F5A	0.4567(12)	0.5095(13)	0.7804(8)	0.082(5)
F6A	0.4346(10)	0.4230(15)	0.8643(11)	0.101(5)
F7A	0.5218(12)	0.4988(13)	0.8852(9)	0.083(5)
F8A	0.5073(10)	0.3791(10)	0.8110(14)	0.061(4)
N1S	0.58829(15)	0.7231(2)	0.79656(16)	0.0454(8)
C1S	0.53885(18)	0.7248(2)	0.79960(17)	0.0356(8)
C2S	0.47436(17)	0.7259(3)	0.80277(19)	0.0455(9)

Table S10. Bond Angles for **6**

Re1-C24	1.864(3)	Re1-N1	1.933(2)
Re1-N3	1.942(2)	Re1-N4	2.139(2)
Re1-N5	2.152(2)	Re1-N2	2.244(2)
O1-C24	1.168(3)	N1-C6	1.440(3)
N1-C1	1.477(3)	N2-C2	1.485(3)
N2-C5	1.488(3)	N2-C3	1.500(3)
N3-C15	1.437(3)	N3-C4	1.489(3)
N4-C25	1.134(3)	N5-C27	1.142(3)
C1-C2	1.513(4)	C1-H1A	0.99
C1-H1B	0.99	C2-H2A	0.99
C2-H2B	0.99	C3-C4	1.515(4)
C3-H3A	0.99	C3-H3B	0.99
C4-H4A	0.99	C4-H4B	0.99
C5-H5A	0.98	C5-H5B	0.98
C5-H5C	0.98	C6-C7	1.402(4)
C6-C11	1.408(4)	C7-C8	1.394(4)
C7-C12	1.510(4)	C8-C9	1.389(4)
C8-H8	0.95	C9-C10	1.381(4)
C9-C13	1.509(4)	C10-C11	1.394(4)
C10-H10	0.95	C11-C14	1.508(4)
C12-H12A	0.98	C12-H12B	0.98
C12-H12C	0.98	C13-H13A	0.98
C13-H13B	0.98	C13-H13C	0.98
C14-H14A	0.98	C14-H14B	0.98
C14-H14C	0.98	C15-C16	1.405(3)
C15-C20	1.411(3)	C16-C17	1.393(4)
C16-C21	1.501(3)	C17-C18	1.388(4)
C17-H17	0.95	C18-C19	1.384(4)
C18-C22	1.510(4)	C19-C20	1.387(4)
C19-H19	0.95	C20-C23	1.511(4)
C21-H21A	0.98	C21-H21B	0.98
C21-H21C	0.98	C22-H22A	0.98
C22-H22B	0.98	C22-H22C	0.98
C23-H23A	0.98	C23-H23B	0.98
C23-H23C	0.98	C25-C26	1.452(4)
C26-H26A	0.98	C26-H26B	0.98
C26-H26C	0.98	C27-C28	1.455(4)
C28-H28A	0.98	C28-H28B	0.98
C28-H28C	0.98	Re2-C52	1.867(3)

Re2-N6	1.934(2)	Re2-N8	1.940(2)
Re2-N9	2.158(2)	Re2-N10	2.166(2)
Re2-N7	2.250(2)	O2-C52	1.168(3)
N6-C34	1.445(3)	N6-C29	1.474(3)
N7-C33	1.485(3)	N7-C30	1.487(4)
N7-C31	1.505(3)	N8-C43	1.434(3)
N8-C32	1.484(3)	N9-C53	1.132(3)
N10-C55	1.140(3)	C29-C30	1.515(4)
C29-H29A	0.99	C29-H29B	0.99
C30-H30A	0.99	C30-H30B	0.99
C31-C32	1.511(4)	C31-H31A	0.99
C31-H31B	0.99	C32-H32A	0.99
C32-H32B	0.99	C33-H33A	0.98
C33-H33B	0.98	C33-H33C	0.98
C34-C35	1.403(4)	C34-C39	1.407(4)
C35-C36	1.395(4)	C35-C40	1.511(4)
C36-C37	1.386(4)	C36-H36	0.95
C37-C38	1.382(4)	C37-C41	1.515(4)
C38-C39	1.395(4)	C38-H38	0.95
C39-C42	1.505(4)	C40-H40A	0.98
C40-H40B	0.98	C40-H40C	0.98
C41-H41A	0.98	C41-H41B	0.98
C41-H41C	0.98	C42-H42A	0.98
C42-H42B	0.98	C42-H42C	0.98
C43-C44	1.403(3)	C43-C48	1.406(3)
C44-C45	1.393(4)	C44-C49	1.504(3)
C45-C46	1.395(4)	C45-H45	0.95
C46-C47	1.383(4)	C46-C50	1.501(4)
C47-C48	1.390(4)	C47-H47	0.95
C48-C51	1.510(3)	C49-H49A	0.98
C49-H49B	0.98	C49-H49C	0.98
C50-H50A	0.98	C50-H50B	0.98
C50-H50C	0.98	C51-H51A	0.98
C51-H51B	0.98	C51-H51C	0.98
C53-C54	1.458(4)	C54-H54A	0.98
C54-H54B	0.98	C54-H54C	0.98
C55-C56	1.454(4)	C56-H56A	0.98
C56-H56B	0.98	C56-H56C	0.98
B1-F3	1.381(4)	B1-F4	1.386(4)
B1-F2	1.388(4)	B1-F1	1.406(4)
B2-F6	1.354(7)	B2-F5	1.361(7)

B2-F8	1.386(8)	B2-F7	1.388(8)
B2A-F6A	1.344(13)	B2A-F5A	1.355(13)
B2A-F7A	1.380(12)	B2A-F8A	1.391(13)
N1S-C1S	1.123(5)	C1S-C2S	1.460(5)
C2S-H2SA	0.98	C2S-H2SB	0.98
C2S-H2SC	0.98		

Table S11. Bond Angles for **4b**

C24-Re1-N1	93.44(10)	C24-Re1-N3	96.43(9)
N1-Re1-N3	108.48(9)	C24-Re1-N4	91.38(9)
N1-Re1-N4	88.32(9)	N3-Re1-N4	160.92(8)
C24-Re1-N5	98.66(9)	N1-Re1-N5	159.41(9)
N3-Re1-N5	86.74(8)	N4-Re1-N5	74.86(8)
C24-Re1-N2	173.48(9)	N1-Re1-N2	81.13(8)
N3-Re1-N2	81.99(8)	N4-Re1-N2	92.06(8)
N5-Re1-N2	87.58(8)	C6-N1-C1	113.5(2)
C6-N1-Re1	128.71(16)	C1-N1-Re1	117.71(16)
C2-N2-C5	108.7(2)	C2-N2-C3	110.6(2)
C5-N2-C3	108.8(2)	C2-N2-Re1	105.80(15)
C5-N2-Re1	117.32(16)	C3-N2-Re1	105.51(14)
C15-N3-C4	113.63(19)	C15-N3-Re1	132.17(16)
C4-N3-Re1	114.05(15)	C25-N4-Re1	174.6(2)
C27-N5-Re1	171.6(2)	N1-C1-C2	109.4(2)
N1-C1-H1A	109.8	C2-C1-H1A	109.8
N1-C1-H1B	109.8	C2-C1-H1B	109.8
H1A-C1-H1B	108.2	N2-C2-C1	110.4(2)
N2-C2-H2A	109.6	C1-C2-H2A	109.6
N2-C2-H2B	109.6	C1-C2-H2B	109.6
H2A-C2-H2B	108.1	N2-C3-C4	109.6(2)
N2-C3-H3A	109.8	C4-C3-H3A	109.8
N2-C3-H3B	109.8	C4-C3-H3B	109.8
H3A-C3-H3B	108.2	N3-C4-C3	108.3(2)
N3-C4-H4A	110.0	C3-C4-H4A	110.0
N3-C4-H4B	110.0	C3-C4-H4B	110.0
H4A-C4-H4B	108.4	N2-C5-H5A	109.5
N2-C5-H5B	109.5	H5A-C5-H5B	109.5
N2-C5-H5C	109.5	H5A-C5-H5C	109.5
H5B-C5-H5C	109.5	C7-C6-C11	119.9(2)
C7-C6-N1	120.2(2)	C11-C6-N1	119.9(2)
C8-C7-C6	118.9(2)	C8-C7-C12	118.6(2)
C6-C7-C12	122.5(2)	C9-C8-C7	122.2(3)
C9-C8-H8	118.9	C7-C8-H8	118.9
C10-C9-C8	117.9(2)	C10-C9-C13	121.1(3)
C8-C9-C13	120.9(3)	C9-C10-C11	122.3(3)
C9-C10-H10	118.8	C11-C10-H10	118.8
C10-C11-C6	118.8(2)	C10-C11-C14	118.0(2)
C6-C11-C14	123.2(2)	C7-C12-H12A	109.5

C7-C12-H12B	109.5	H12A-C12-H12B	109.5
C7-C12-H12C	109.5	H12A-C12-H12C	109.5
H12B-C12-H12C	109.5	C9-C13-H13A	109.5
C9-C13-H13B	109.5	H13A-C13-H13B	109.5
C9-C13-H13C	109.5	H13A-C13-H13C	109.5
H13B-C13-H13C	109.5	C11-C14-H14A	109.5
C11-C14-H14B	109.5	H14A-C14-H14B	109.5
C11-C14-H14C	109.5	H14A-C14-H14C	109.5
H14B-C14-H14C	109.5	C16-C15-C20	119.3(2)
C16-C15-N3	121.6(2)	C20-C15-N3	119.0(2)
C17-C16-C15	119.0(2)	C17-C16-C21	117.7(2)
C15-C16-C21	123.2(2)	C18-C17-C16	122.4(2)
C18-C17-H17	118.8	C16-C17-H17	118.8
C19-C18-C17	117.5(2)	C19-C18-C22	121.9(3)
C17-C18-C22	120.6(3)	C18-C19-C20	122.6(2)
C18-C19-H19	118.7	C20-C19-H19	118.7
C19-C20-C15	119.1(2)	C19-C20-C23	118.7(2)
C15-C20-C23	122.2(2)	C16-C21-H21A	109.5
C16-C21-H21B	109.5	H21A-C21-H21B	109.5
C16-C21-H21C	109.5	H21A-C21-H21C	109.5
H21B-C21-H21C	109.5	C18-C22-H22A	109.5
C18-C22-H22B	109.5	H22A-C22-H22B	109.5
C18-C22-H22C	109.5	H22A-C22-H22C	109.5
H22B-C22-H22C	109.5	C20-C23-H23A	109.5
C20-C23-H23B	109.5	H23A-C23-H23B	109.5
C20-C23-H23C	109.5	H23A-C23-H23C	109.5
H23B-C23-H23C	109.5	O1-C24-Re1	177.2(2)
N4-C25-C26	177.6(3)	C25-C26-H26A	109.5
C25-C26-H26B	109.5	H26A-C26-H26B	109.5
C25-C26-H26C	109.5	H26A-C26-H26C	109.5
H26B-C26-H26C	109.5	N5-C27-C28	177.7(3)
C27-C28-H28A	109.5	C27-C28-H28B	109.5
H28A-C28-H28B	109.5	C27-C28-H28C	109.5
H28A-C28-H28C	109.5	H28B-C28-H28C	109.5
C52-Re2-N6	94.34(10)	C52-Re2-N8	96.19(10)
N6-Re2-N8	107.31(9)	C52-Re2-N9	91.78(9)
N6-Re2-N9	88.87(8)	N8-Re2-N9	161.28(9)
C52-Re2-N10	97.35(9)	N6-Re2-N10	160.43(9)
N8-Re2-N10	87.00(8)	N9-Re2-N10	75.18(8)
C52-Re2-N7	173.86(9)	N6-Re2-N7	81.05(8)
N8-Re2-N7	81.43(8)	N9-Re2-N7	92.17(8)

N10-Re2-N7	88.20(8)	C34-N6-C29	113.3(2)
C34-N6-Re2	129.26(16)	C29-N6-Re2	117.33(17)
C33-N7-C30	109.0(2)	C33-N7-C31	109.3(2)
C30-N7-C31	110.6(2)	C33-N7-Re2	116.08(16)
C30-N7-Re2	105.53(15)	C31-N7-Re2	106.15(15)
C43-N8-C32	113.48(19)	C43-N8-Re2	131.83(16)
C32-N8-Re2	114.62(16)	C53-N9-Re2	174.4(2)
C55-N10-Re2	175.8(2)	N6-C29-C30	109.4(2)
N6-C29-H29A	109.8	C30-C29-H29A	109.8
N6-C29-H29B	109.8	C30-C29-H29B	109.8
H29A-C29-H29B	108.2	N7-C30-C29	109.6(2)
N7-C30-H30A	109.8	C29-C30-H30A	109.8
N7-C30-H30B	109.8	C29-C30-H30B	109.8
H30A-C30-H30B	108.2	N7-C31-C32	109.2(2)
N7-C31-H31A	109.8	C32-C31-H31A	109.8
N7-C31-H31B	109.8	C32-C31-H31B	109.8
H31A-C31-H31B	108.3	N8-C32-C31	108.8(2)
N8-C32-H32A	109.9	C31-C32-H32A	109.9
N8-C32-H32B	109.9	C31-C32-H32B	109.9
H32A-C32-H32B	108.3	N7-C33-H33A	109.5
N7-C33-H33B	109.5	H33A-C33-H33B	109.5
N7-C33-H33C	109.5	H33A-C33-H33C	109.5
H33B-C33-H33C	109.5	C35-C34-C39	119.9(2)
C35-C34-N6	121.1(2)	C39-C34-N6	119.0(2)
C36-C35-C34	119.0(3)	C36-C35-C40	117.9(3)
C34-C35-C40	123.0(2)	C37-C36-C35	121.9(3)
C37-C36-H36	119.0	C35-C36-H36	119.0
C38-C37-C36	118.1(3)	C38-C37-C41	120.3(3)
C36-C37-C41	121.5(3)	C37-C38-C39	122.3(3)
C37-C38-H38	118.8	C39-C38-H38	118.8
C38-C39-C34	118.6(3)	C38-C39-C42	118.0(2)
C34-C39-C42	123.4(2)	C35-C40-H40A	109.5
C35-C40-H40B	109.5	H40A-C40-H40B	109.5
C35-C40-H40C	109.5	H40A-C40-H40C	109.5
H40B-C40-H40C	109.5	C37-C41-H41A	109.5
C37-C41-H41B	109.5	H41A-C41-H41B	109.5
C37-C41-H41C	109.5	H41A-C41-H41C	109.5
H41B-C41-H41C	109.5	C39-C42-H42A	109.5
C39-C42-H42B	109.5	H42A-C42-H42B	109.5
C39-C42-H42C	109.5	H42A-C42-H42C	109.5
H42B-C42-H42C	109.5	C44-C43-C48	119.5(2)

C44-C43-N8	121.8(2)	C48-C43-N8	118.7(2)
C45-C44-C43	119.1(2)	C45-C44-C49	118.7(2)
C43-C44-C49	122.0(2)	C44-C45-C46	122.3(2)
C44-C45-H45	118.8	C46-C45-H45	118.8
C47-C46-C45	117.1(2)	C47-C46-C50	121.5(2)
C45-C46-C50	121.2(2)	C46-C47-C48	122.8(2)
C46-C47-H47	118.6	C48-C47-H47	118.6
C47-C48-C43	119.0(2)	C47-C48-C51	118.7(2)
C43-C48-C51	122.2(2)	C44-C49-H49A	109.5
C44-C49-H49B	109.5	H49A-C49-H49B	109.5
C44-C49-H49C	109.5	H49A-C49-H49C	109.5
H49B-C49-H49C	109.5	C46-C50-H50A	109.5
C46-C50-H50B	109.5	H50A-C50-H50B	109.5
C46-C50-H50C	109.5	H50A-C50-H50C	109.5
H50B-C50-H50C	109.5	C48-C51-H51A	109.5
C48-C51-H51B	109.5	H51A-C51-H51B	109.5
C48-C51-H51C	109.5	H51A-C51-H51C	109.5
H51B-C51-H51C	109.5	O2-C52-Re2	178.5(2)
N9-C53-C54	178.5(3)	C53-C54-H54A	109.5
C53-C54-H54B	109.5	H54A-C54-H54B	109.5
C53-C54-H54C	109.5	H54A-C54-H54C	109.5
H54B-C54-H54C	109.5	N10-C55-C56	178.9(3)
C55-C56-H56A	109.5	C55-C56-H56B	109.5
H56A-C56-H56B	109.5	C55-C56-H56C	109.5
H56A-C56-H56C	109.5	H56B-C56-H56C	109.5
F3-B1-F4	110.3(3)	F3-B1-F2	109.8(3)
F4-B1-F2	109.6(3)	F3-B1-F1	109.5(3)
F4-B1-F1	108.5(3)	F2-B1-F1	109.2(3)
F6-B2-F5	115.1(7)	F6-B2-F8	108.7(7)
F5-B2-F8	111.0(7)	F6-B2-F7	107.7(7)
F5-B2-F7	107.8(7)	F8-B2-F7	106.1(7)
F6A-B2A-F5A	111.9(12)	F6A-B2A-F7A	107.8(11)
F5A-B2A-F7A	108.5(11)	F6A-B2A-F8A	109.7(11)
F5A-B2A-F8A	109.3(11)	F7A-B2A-F8A	109.5(11)
N1S-C1S-C2S	179.1(4)	C1S-C2S-H2SA	109.5
C1S-C2S-H2SB	109.5	H2SA-C2S-H2SB	109.5
C1S-C2S-H2SC	109.5	H2SA-C2S-H2SC	109.5
H2SB-C2S-H2SC	109.5		

Table S12. Torsion Angles for **4b**

C6-N1-C1-C2	-146.8(2)	Re1-N1-C1-C2	30.9(3)
C5-N2-C2-C1	162.4(2)	C3-N2-C2-C1	-78.2(3)
Re1-N2-C2-C1	35.6(2)	N1-C1-C2-N2	-43.8(3)
C2-N2-C3-C4	147.1(2)	C5-N2-C3-C4	-93.6(2)
Re1-N2-C3-C4	33.2(2)	C15-N3-C4-C3	-140.8(2)
Re1-N3-C4-C3	43.1(2)	N2-C3-C4-N3	-49.8(3)
C1-N1-C6-C7	-104.3(3)	Re1-N1-C6-C7	78.3(3)
C1-N1-C6-C11	74.0(3)	Re1-N1-C6-C11	-103.4(3)
C11-C6-C7-C8	1.2(4)	N1-C6-C7-C8	179.5(2)
C11-C6-C7-C12	-176.3(2)	N1-C6-C7-C12	2.0(4)
C6-C7-C8-C9	-0.8(4)	C12-C7-C8-C9	176.8(3)
C7-C8-C9-C10	0.4(4)	C7-C8-C9-C13	-177.8(3)
C8-C9-C10-C11	-0.4(4)	C13-C9-C10-C11	177.8(3)
C9-C10-C11-C6	0.8(4)	C9-C10-C11-C14	-176.6(2)
C7-C6-C11-C10	-1.2(4)	N1-C6-C11-C10	-179.5(2)
C7-C6-C11-C14	176.0(2)	N1-C6-C11-C14	-2.3(4)
C4-N3-C15-C16	-109.5(3)	Re1-N3-C15-C16	65.7(3)
C4-N3-C15-C20	69.3(3)	Re1-N3-C15-C20	-115.4(2)
C20-C15-C16-C17	2.4(4)	N3-C15-C16-C17	-178.8(2)
C20-C15-C16-C21	-173.1(2)	N3-C15-C16-C21	5.7(4)
C15-C16-C17-C18	-2.9(4)	C21-C16-C17-C18	172.8(2)
C16-C17-C18-C19	1.4(4)	C16-C17-C18-C22	-176.9(2)
C17-C18-C19-C20	0.7(4)	C22-C18-C19-C20	178.9(2)
C18-C19-C20-C15	-1.1(4)	C18-C19-C20-C23	179.0(2)
C16-C15-C20-C19	-0.4(4)	N3-C15-C20-C19	-179.3(2)
C16-C15-C20-C23	179.4(2)	N3-C15-C20-C23	0.6(4)
C34-N6-C29-C30	-144.4(2)	Re2-N6-C29-C30	32.6(3)
C33-N7-C30-C29	162.2(2)	C31-N7-C30-C29	-77.5(3)
Re2-N7-C30-C29	36.9(2)	N6-C29-C30-N7	-45.9(3)
C33-N7-C31-C32	-94.0(2)	C30-N7-C31-C32	145.9(2)
Re2-N7-C31-C32	31.9(2)	C43-N8-C32-C31	-139.6(2)
Re2-N8-C32-C31	43.2(2)	N7-C31-C32-N8	-48.6(3)
C29-N6-C34-C35	-109.3(3)	Re2-N6-C34-C35	74.1(3)
C29-N6-C34-C39	69.5(3)	Re2-N6-C34-C39	-107.1(3)
C39-C34-C35-C36	1.1(4)	N6-C34-C35-C36	179.9(2)
C39-C34-C35-C40	-175.0(2)	N6-C34-C35-C40	3.8(4)
C34-C35-C36-C37	-1.2(4)	C40-C35-C36-C37	175.2(2)
C35-C36-C37-C38	1.2(4)	C35-C36-C37-C41	-178.1(3)
C36-C37-C38-C39	-1.3(4)	C41-C37-C38-C39	178.1(2)

C37-C38-C39-C34	1.3(4)	C37-C38-C39-C42	-177.2(2)
C35-C34-C39-C38	-1.2(4)	N6-C34-C39-C38	180.0(2)
C35-C34-C39-C42	177.2(2)	N6-C34-C39-C42	-1.6(4)
C32-N8-C43-C44	-106.3(3)	Re2-N8-C43-C44	70.3(3)
C32-N8-C43-C48	71.6(3)	Re2-N8-C43-C48	-111.8(2)
C48-C43-C44-C45	2.6(4)	N8-C43-C44-C45	-179.5(2)
C48-C43-C44-C49	-172.5(2)	N8-C43-C44-C49	5.4(4)
C43-C44-C45-C46	-3.5(4)	C49-C44-C45-C46	171.8(2)
C44-C45-C46-C47	1.7(4)	C44-C45-C46-C50	-175.1(2)
C45-C46-C47-C48	0.8(4)	C50-C46-C47-C48	177.6(2)
C46-C47-C48-C43	-1.5(4)	C46-C47-C48-C51	-179.8(2)
C44-C43-C48-C47	-0.3(4)	N8-C43-C48-C47	-178.2(2)
C44-C43-C48-C51	178.0(2)	N8-C43-C48-C51	0.0(4)

Table S13. Anisotropic atomic displacement parameters (Å^2) for **4b**

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Re1	0.01207(5)	0.00893(4)	0.01124(5)	0.00030(3)	0.00264(4)	-0.00084(3)
O1	0.0236(10)	0.0131(9)	0.0193(10)	0.0014(7)	0.0050(8)	0.0049(7)
N1	0.0154(11)	0.0122(10)	0.0141(11)	0.0004(8)	0.0057(9)	-0.0026(8)
N2	0.0142(11)	0.0156(10)	0.0127(11)	0.0015(8)	0.0006(9)	0.0011(8)
N3	0.0143(10)	0.0104(10)	0.0106(10)	-0.0009(8)	0.0026(8)	-0.0008(8)
N4	0.0178(11)	0.0140(11)	0.0169(12)	-0.0006(8)	0.0051(10)	-0.0011(8)
N5	0.0170(11)	0.0139(10)	0.0138(11)	-0.0010(8)	0.0014(9)	-0.0004(8)
C1	0.0274(15)	0.0129(12)	0.0195(14)	0.0047(10)	0.0111(12)	-0.0014(10)
C2	0.0288(15)	0.0151(13)	0.0163(14)	0.0038(10)	0.0043(12)	0.0028(10)
C3	0.0206(14)	0.0110(12)	0.0199(14)	-0.0011(10)	0.0024(11)	0.0017(9)
C4	0.0185(13)	0.0119(12)	0.0169(13)	-0.0023(10)	0.0027(11)	0.0024(9)
C5	0.0181(14)	0.0182(14)	0.0269(16)	0.0021(11)	-0.0006(12)	0.0058(10)
C6	0.0183(13)	0.0104(12)	0.0147(13)	0.0021(9)	0.0071(10)	-0.0033(9)
C7	0.0198(14)	0.0151(12)	0.0174(14)	-0.0006(10)	0.0068(11)	-0.0035(10)
C8	0.0154(13)	0.0193(14)	0.0258(16)	0.0022(11)	0.0065(12)	-0.0002(10)
C9	0.0244(15)	0.0137(13)	0.0233(15)	0.0001(10)	0.0117(12)	-0.0006(10)
C10	0.0233(14)	0.0163(13)	0.0183(14)	-0.0026(10)	0.0089(12)	-0.0039(10)
C11	0.0222(14)	0.0127(12)	0.0149(13)	0.0017(10)	0.0077(11)	-0.0034(10)
C12	0.0203(15)	0.0358(17)	0.0216(16)	-0.0082(12)	0.0006(12)	0.0003(12)
C13	0.0294(17)	0.0292(16)	0.0302(18)	-0.0074(13)	0.0137(14)	0.0039(12)
C14	0.0267(15)	0.0195(14)	0.0135(13)	0.0010(10)	0.0035(12)	0.0003(11)
C15	0.0109(12)	0.0132(12)	0.0124(12)	-0.0030(9)	0.0039(10)	0.0006(9)
C16	0.0145(12)	0.0144(12)	0.0136(13)	-0.0021(9)	0.0050(10)	0.0021(9)
C17	0.0203(13)	0.0185(13)	0.0145(13)	0.0013(10)	0.0093(11)	0.0053(10)
C18	0.0167(13)	0.0237(14)	0.0137(13)	-0.0029(10)	0.0019(11)	0.0084(10)
C19	0.0123(12)	0.0206(14)	0.0194(14)	-0.0069(11)	0.0029(11)	0.0002(10)
C20	0.0149(12)	0.0158(12)	0.0160(13)	-0.0030(10)	0.0046(10)	-0.0001(9)
C21	0.0201(14)	0.0173(13)	0.0145(13)	0.0022(10)	0.0057(11)	-0.0013(10)
C22	0.0244(15)	0.0312(17)	0.0196(15)	-0.0011(12)	0.0006(12)	0.0044(12)
C23	0.0187(14)	0.0177(13)	0.0224(15)	-0.0005(11)	0.0026(12)	-0.0056(10)
C24	0.0171(13)	0.0135(12)	0.0115(12)	-0.0048(9)	0.0061(10)	-0.0059(9)
C25	0.0191(13)	0.0156(13)	0.0210(15)	0.0006(10)	0.0076(12)	0.0013(10)
C26	0.0206(15)	0.0284(16)	0.0227(16)	-0.0098(12)	-0.0019(12)	-0.0007(11)
C27	0.0171(13)	0.0180(13)	0.0118(13)	-0.0002(10)	-0.0002(10)	-0.0011(10)
C28	0.0134(13)	0.0349(16)	0.0199(15)	-0.0026(12)	0.0036(11)	-0.0019(11)
Re2	0.01486(5)	0.00980(5)	0.01108(5)	-0.00157(4)	0.00483(4)	-0.00120(3)
O2	0.0229(10)	0.0135(9)	0.0263(11)	0.0016(8)	0.0071(9)	0.0015(7)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N6	0.0187(11)	0.0117(10)	0.0138(11)	-0.0019(8)	0.0060(9)	-0.0021(8)
N7	0.0219(12)	0.0125(10)	0.0164(12)	-0.0003(8)	0.0039(10)	0.0026(8)
N8	0.0185(11)	0.0110(10)	0.0125(11)	-0.0022(8)	0.0054(9)	0.0001(8)
N9	0.0206(12)	0.0146(11)	0.0154(12)	-0.0006(9)	0.0069(10)	-0.0004(8)
N10	0.0176(11)	0.0161(11)	0.0136(11)	-0.0018(8)	0.0030(9)	-0.0018(8)
C29	0.0303(15)	0.0133(13)	0.0161(14)	0.0008(10)	0.0090(12)	-0.0021(10)
C30	0.0309(16)	0.0184(14)	0.0165(14)	0.0036(10)	0.0064(12)	0.0011(11)
C31	0.0221(14)	0.0099(12)	0.0224(15)	-0.0022(10)	0.0051(12)	0.0032(10)
C32	0.0188(13)	0.0126(12)	0.0194(14)	-0.0037(10)	0.0059(11)	0.0019(9)
C33	0.0218(14)	0.0206(14)	0.0245(16)	-0.0012(11)	-0.0009(12)	0.0050(11)
C34	0.0222(14)	0.0102(11)	0.0147(13)	-0.0028(9)	0.0088(11)	-0.0044(9)
C35	0.0214(14)	0.0146(13)	0.0202(14)	-0.0028(10)	0.0108(12)	-0.0039(10)
C36	0.0228(15)	0.0187(14)	0.0266(16)	0.0015(11)	0.0126(12)	-0.0022(10)
C37	0.0347(17)	0.0120(13)	0.0328(17)	-0.0039(11)	0.0235(14)	-0.0055(11)
C38	0.0343(17)	0.0167(13)	0.0183(14)	-0.0036(11)	0.0155(13)	-0.0097(11)
C39	0.0282(15)	0.0131(12)	0.0158(14)	-0.0017(10)	0.0115(12)	-0.0083(10)
C40	0.0215(14)	0.0236(14)	0.0202(15)	-0.0067(11)	0.0050(12)	-0.0004(11)
C41	0.041(2)	0.0236(16)	0.048(2)	-0.0076(14)	0.0308(17)	-0.0036(13)
C42	0.0366(17)	0.0204(14)	0.0140(14)	-0.0038(11)	0.0097(13)	-0.0066(12)
C43	0.0165(12)	0.0103(12)	0.0129(12)	-0.0037(9)	0.0067(10)	0.0006(9)
C44	0.0172(12)	0.0124(12)	0.0134(12)	-0.0045(9)	0.0072(10)	-0.0002(9)
C45	0.0195(13)	0.0135(12)	0.0145(13)	-0.0007(10)	0.0076(11)	0.0018(9)
C46	0.0136(12)	0.0160(12)	0.0182(14)	-0.0033(10)	0.0056(11)	0.0016(9)
C47	0.0160(13)	0.0142(13)	0.0194(14)	-0.0051(10)	0.0083(11)	-0.0008(9)
C48	0.0177(13)	0.0126(12)	0.0144(13)	-0.0035(9)	0.0080(10)	-0.0008(9)
C49	0.0233(14)	0.0180(13)	0.0187(14)	0.0001(10)	0.0075(12)	-0.0057(10)
C50	0.0184(13)	0.0224(14)	0.0206(15)	-0.0033(11)	0.0042(11)	0.0004(10)
C51	0.0208(14)	0.0178(13)	0.0195(14)	-0.0002(10)	0.0089(12)	-0.0051(10)
C52	0.0176(13)	0.0130(12)	0.0124(12)	-0.0047(9)	0.0079(10)	-0.0048(9)
C53	0.0178(13)	0.0171(13)	0.0160(13)	0.0000(10)	0.0056(11)	0.0029(10)
C54	0.0193(15)	0.0317(16)	0.0232(16)	-0.0131(12)	-0.0030(12)	0.0013(12)
C55	0.0205(14)	0.0177(13)	0.0173(14)	-0.0028(10)	0.0044(11)	0.0003(10)
C56	0.0217(15)	0.0360(18)	0.0345(19)	-0.0066(14)	0.0175(14)	-0.0038(12)
B1	0.0209(17)	0.0331(19)	0.0181(17)	-0.0017(13)	0.0039(14)	-0.0002(13)
F1	0.0272(10)	0.0333(10)	0.0306(10)	-0.0053(8)	0.0073(8)	-0.0038(7)
F2	0.0325(10)	0.0466(11)	0.0247(10)	-0.0008(8)	0.0114(8)	-0.0024(8)
F3	0.0384(12)	0.0542(13)	0.0434(13)	-0.0282(10)	0.0168(10)	-0.0133(9)
F4	0.0290(11)	0.0746(16)	0.0592(15)	0.0338(12)	0.0075(11)	0.0112(10)
B2	0.025(4)	0.029(4)	0.011(3)	-0.009(2)	0.004(3)	-0.013(3)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F5	0.043(4)	0.067(5)	0.018(3)	0.004(3)	0.000(2)	-0.003(3)
F6	0.055(4)	0.084(5)	0.039(4)	-0.011(3)	0.033(3)	-0.020(4)
F7	0.075(4)	0.046(4)	0.024(3)	-0.004(2)	-0.009(3)	-0.040(3)
F8	0.062(4)	0.041(3)	0.066(6)	-0.004(3)	0.029(4)	0.003(3)
B2A	0.061(8)	0.044(6)	0.055(8)	-0.018(5)	0.015(6)	-0.023(5)
F5A	0.128(11)	0.051(5)	0.057(6)	-0.013(4)	0.000(6)	-0.002(6)
F6A	0.082(8)	0.134(9)	0.100(10)	-0.022(7)	0.045(7)	-0.062(6)
F7A	0.113(9)	0.057(6)	0.066(7)	-0.008(5)	-0.001(5)	-0.040(6)
F8A	0.076(7)	0.037(4)	0.080(9)	-0.017(5)	0.038(7)	-0.028(4)
N1S	0.0370(18)	0.051(2)	0.0396(19)	-0.0023(15)	-0.0073(15)	0.0059(14)
C1S	0.046(2)	0.0356(19)	0.0179(16)	-0.0055(13)	-0.0054(16)	0.0047(16)
C2S	0.050(2)	0.054(2)	0.034(2)	-0.0058(17)	0.0120(19)	0.0027(18)

Table S14. Hydrogen atomic coordinates and isotropic displacement parameters ($Å^2$) for **4b**

	x	y	z	U(eq)
H1A	0.2112	0.1478	0.5173	0.023
H1B	0.2331	0.1952	0.5927	0.023
H2A	0.3349	0.1850	0.5922	0.024
H2B	0.3071	0.0886	0.5632	0.024
H3A	0.2584	0.0790	0.4414	0.021
H3B	0.3257	0.0486	0.4353	0.021
H4A	0.3251	0.1546	0.3476	0.019
H4B	0.2604	0.1022	0.3251	0.019
H5A	0.4072	0.1609	0.4583	0.033
H5B	0.4070	0.1046	0.5278	0.033
H5C	0.4133	0.2126	0.5312	0.033
H8	0.0574	0.4284	0.4904	0.024
H10	0.1862	0.4657	0.6700	0.022
H12A	0.1435	0.2736	0.4132	0.04
H12B	0.1000	0.3592	0.3873	0.04
H12C	0.0753	0.2762	0.4251	0.04
H13A	0.0703	0.5770	0.5900	0.043
H13B	0.0852	0.5223	0.6624	0.043
H13C	0.0298	0.4904	0.5989	0.043
H14A	0.2683	0.3254	0.6927	0.03
H14B	0.2956	0.4176	0.6700	0.03
H14C	0.3008	0.3257	0.6288	0.03
H17	0.1669	0.3774	0.1612	0.02
H19	0.0704	0.1715	0.2146	0.021
H21A	0.2657	0.4058	0.2156	0.026
H21B	0.3044	0.3280	0.2636	0.026
H21C	0.2753	0.4091	0.2989	0.026
H22A	0.0647	0.2455	0.0760	0.039
H22B	0.0659	0.3518	0.0919	0.039
H22C	0.0204	0.2870	0.1208	0.039
H23A	0.0979	0.1125	0.3315	0.03
H23B	0.1641	0.1334	0.3820	0.03
H23C	0.1576	0.0614	0.3198	0.03
H26A	0.4246	0.4314	0.6692	0.038
H26B	0.4257	0.5183	0.6210	0.038
H26C	0.4717	0.4358	0.6198	0.038
H28A	0.5018	0.3583	0.4157	0.034

	x	y	z	U(eq)
H28B	0.4689	0.3775	0.3352	0.034
H28C	0.4832	0.2757	0.3620	0.034
H29A	0.1799	0.6664	0.6222	0.023
H29B	0.1944	0.7150	0.6973	0.023
H30A	0.2986	0.7180	0.7048	0.026
H30B	0.2773	0.6179	0.6777	0.026
H31A	0.2343	0.5996	0.5549	0.022
H31B	0.3039	0.5775	0.5523	0.022
H32A	0.3004	0.6814	0.4633	0.02
H32B	0.2383	0.6231	0.4398	0.02
H33A	0.3792	0.6964	0.5773	0.035
H33B	0.3792	0.6463	0.6494	0.035
H33C	0.3791	0.7545	0.6460	0.035
H36	0.0167	0.9456	0.5863	0.026
H38	0.1352	0.9717	0.7749	0.026
H40A	0.1118	0.8132	0.5066	0.033
H40B	0.0549	0.8821	0.4886	0.033
H40C	0.0474	0.7857	0.5227	0.033
H41A	-0.0132	1.0364	0.6716	0.051
H41B	0.0418	1.0815	0.7291	0.051
H41C	0.0090	0.9917	0.7477	0.051
H42A	0.2214	0.8202	0.7901	0.035
H42B	0.2403	0.9248	0.7914	0.035
H42C	0.2598	0.8570	0.7370	0.035
H45	0.1374	0.8775	0.2643	0.018
H47	0.0456	0.6725	0.3270	0.019
H49A	0.2443	0.9226	0.4026	0.029
H49B	0.2367	0.9128	0.3198	0.029
H49C	0.2754	0.8392	0.3723	0.029
H50A	0.0324	0.8426	0.1984	0.031
H50B	-0.0049	0.7622	0.2240	0.031
H50C	0.0464	0.7396	0.1822	0.031
H51A	0.1392	0.5774	0.4418	0.028
H51B	0.0734	0.6187	0.4431	0.028
H51C	0.1343	0.6545	0.4973	0.028
H54A	0.3730	0.9813	0.7806	0.039
H54B	0.3837	1.0576	0.7270	0.039
H54C	0.4264	0.9693	0.7400	0.039
H56A	0.4639	0.9072	0.5139	0.043

	x	y	z	U(eq)
H56B	0.4237	0.9210	0.4353	0.043
H56C	0.4464	0.8218	0.4628	0.043
H2SA	0.4510	0.7666	0.7666	0.068
H2SB	0.4715	0.7471	0.8491	0.068
H2SC	0.4573	0.6646	0.7948	0.068

Experimental for $C_{18}H_{12}Cl_2F_{10}N_3ORe$ (**6**)

Data Collection and Processing. The sample **6** was submitted by Jessica Smeltz of the Ison research group at North Carolina State University. The sample was mounted on a Mitegen polyimide micromount with a small amount of Paratone N oil. All X-ray measurements were made on a Bruker-Nonius Kappa Axis X8 Apex2 diffractometer at a temperature of 110 K. The unit cell dimensions were determined from a symmetry constrained fit of 9871 reflections with $4.84° < 2\theta < 62.72°$. The data collection strategy was a number of ω and φ scans which collected data up to 75.82° (2θ). The frame integration was performed using SAINT.[10] The resulting raw data was scaled and absorption corrected using a multi-scan averaging of symmetry equivalent data using SADABS.[11]

Structure Solution and Refinement. The structure was solved by direct methods using the XS program.[12] All non-hydrogen atoms were obtained from the initial solution. The hydrogen atoms were introduced at idealized positions and were allowed to ride on the parent atom. The structural model was fit to the data using full matrix least-squares based on F^2. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation. The structure was refined using the XL program from SHELXTL,[13] graphic plots were produced using the NRCVAX crystallographic program suite. Additional information and other relevant literature references can be found in the reference section of the Facility's Web page (http://www.xray.ncsu.edu).

Acknowledgement
The authors wish to thank the Department of Chemistry of North Carolina State University and the State of North Carolina for funding the purchase of the Apex2 diffractometer.

10 Bruker-Nonius, SAINT version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

11 Bruker-Nonius, SADABS version 2009.9, **2009**, Bruker-Nonius, Madison, WI 53711, USA

12 Bruker-AXS, XS version 2009.9, **2009**, Bruker-AXS, Madison, WI 53711, USA

13 Bruker-AXS, XL version 2009.9, **2009**, Bruker-AXS, Madison, WI 53711, USA

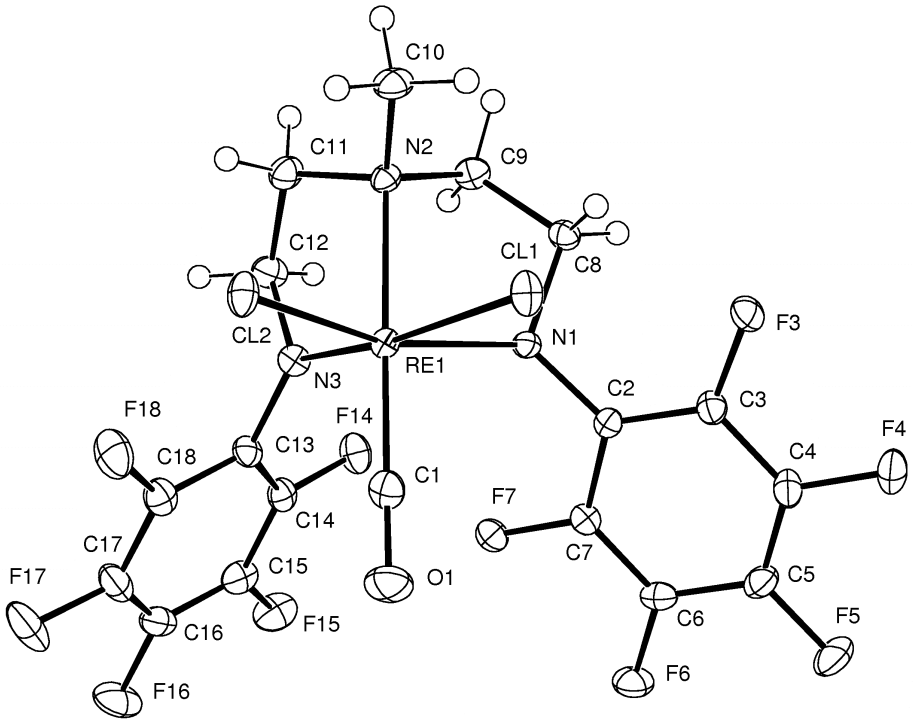


Figure S11. ORTEP drawing of **6** showing naming and numbering scheme. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

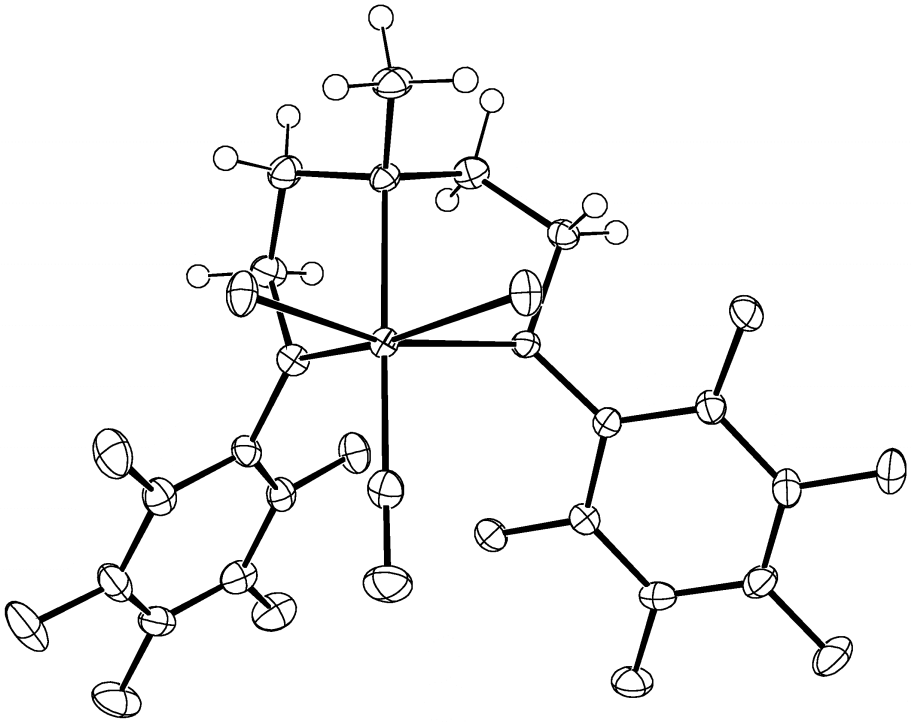


Figure S12. ORTEP drawing of **6**. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

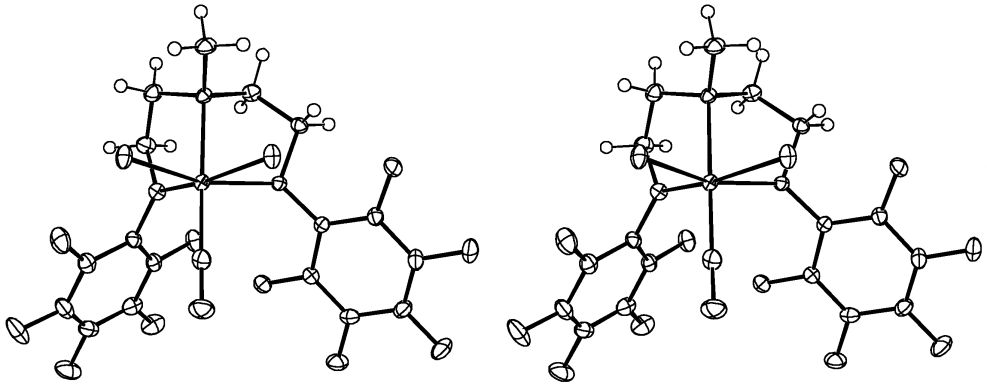


Figure S13. Stereoscopic ORTEP drawing of **6**. Ellipsoids are at the 50% probability level and hydrogen atoms were drawn with arbitrary radii for clarity.

Table S15. Summary of Crystal Data **for 6**

Formula	$C_{18}H_{11}Cl_2F_{10}N_3ORe$
Formula Weight (*g/mol*)	732.40
Crystal Dimensions (*mm*)	0.13 × 0.10 × 0.04
Crystal Color and Habit	green prism
Crystal System	orthorhombic
Space Group	P b c a
Temperature, K	110
a, Å	12.2754(4)
b, Å	18.8021(6)
c, Å	18.9382(6)
α,°	90.00
β,°	90.00
γ,°	90.00
V, Å^3	4371.0(2)
Number of reflections to determine final unit cell	9871
Min and Max 2θ for cell determination, °	4.84, 62.72
Z	8
F(000)	2776
ρ (*g/cm*)	2.226
λ, Å, (MoKα)	0.71073
μ, (*cm*$^{-1}$)	5.907
Diffractometer Type	Bruker-Nonius Kappa Axis X8 Apex2
Scan Type(s)	omega and phi scans
Max 2θ for data collection, °	75.82
Measured fraction of data	0.996
Number of reflections measured	231624
Unique reflections measured	11778
R_{merge}	0.0761
Number of reflections included in refinement	11778
Cut off Threshold Expression	>2sigma(I)
Structure refined using	full matrix least-squares using F^2
Weighting Scheme	calc w=1/[sigma^2(Fo^2)+(0.0285P)^2+4.1132P] where P=(Fo^2+2Fc^2)/3
Number of parameters in least-squares	317
R_1	0.0323
wR_2	0.0609
R_1 (all data)	0.0663
wR_2 (all data)	0.0703
GOF	1.021
Maximum shift/error	0.006
Min & Max peak heights on final ΔF Map (*e*$^-$/Å)	-3.039, 3.349

Where:

$R_1 = \Sigma(|F_o| - |F_c|) / \Sigma F_o$

$wR_2 = [\Sigma(w(F_o^2 - F_c^2)^2) / \Sigma(w F_o^4)]^{½}$

$GOF = [\Sigma(w(F_o^2 - F_c^2)^2) / (\text{No. of reflns.} - \text{No. of params.})]^{½}$

Table S16. Atomic Coordinates for **6**

Atom	x	y	z	$U_{iso/equiv}$
Re1	0.771966(7)	0.786730(5)	0.658621(5)	0.01296(3)
Cl1	0.88517(5)	0.78617(3)	0.76427(3)	0.01889(11)
Cl2	0.92906(5)	0.75536(3)	0.59303(3)	0.02043(11)
C1	0.7948(2)	0.88374(14)	0.64566(13)	0.0199(5)
O1	0.80875(18)	0.94472(10)	0.63811(12)	0.0297(4)
N1	0.64501(16)	0.79359(10)	0.74035(10)	0.0142(3)
N2	0.72678(16)	0.67108(11)	0.67347(10)	0.0149(4)
N3	0.66967(17)	0.77449(11)	0.58290(11)	0.0167(4)
C2	0.63401(18)	0.85616(12)	0.78433(12)	0.0138(4)
C3	0.6609(2)	0.86164(13)	0.85539(12)	0.0170(4)
C4	0.6357(2)	0.92291(14)	0.89356(13)	0.0204(5)
C5	0.5827(2)	0.97881(13)	0.86275(14)	0.0208(5)
C6	0.5571(2)	0.97501(13)	0.79184(14)	0.0186(4)
C7	0.58334(19)	0.91488(12)	0.75374(12)	0.0158(4)
F3	0.70865(13)	0.80894(8)	0.89139(8)	0.0214(3)
F4	0.66134(15)	0.92607(9)	0.96242(8)	0.0298(4)
F5	0.55594(15)	1.03645(8)	0.89999(9)	0.0306(4)
F6	0.50522(13)	1.02884(8)	0.76026(9)	0.0261(3)
F7	0.55593(12)	0.91205(8)	0.68558(8)	0.0205(3)
C8	0.6425(2)	0.72381(12)	0.77929(12)	0.0165(4)
C9	0.6335(2)	0.66656(13)	0.72393(13)	0.0188(4)
C10	0.8157(2)	0.62343(13)	0.69841(14)	0.0215(5)
C11	0.6916(2)	0.64730(13)	0.60226(13)	0.0207(5)
C12	0.6193(2)	0.70362(13)	0.56935(14)	0.0211(5)
C13	0.6313(2)	0.82689(13)	0.53450(12)	0.0166(4)
C14	0.5257(2)	0.85326(13)	0.53851(12)	0.0182(4)
C15	0.4874(2)	0.90384(14)	0.49184(14)	0.0218(5)
C16	0.5541(2)	0.92774(14)	0.43830(14)	0.0243(5)
C17	0.6575(2)	0.90139(15)	0.43170(13)	0.0259(6)
C18	0.6962(2)	0.85167(14)	0.47930(13)	0.0212(5)
F14	0.45804(12)	0.82843(9)	0.58806(8)	0.0237(3)
F15	0.38537(14)	0.92864(9)	0.49822(9)	0.0307(4)
F16	0.51646(17)	0.97623(9)	0.39266(9)	0.0371(4)
F17	0.72083(16)	0.92332(11)	0.37818(9)	0.0385(5)
F18	0.79652(14)	0.82584(10)	0.47019(9)	0.0310(4)
H8A	0.5792	0.7220	0.8117	0.020
H8B	0.7099	0.7175	0.8073	0.020
H9A	0.6333	0.6193	0.7470	0.023
H9B	0.5641	0.6721	0.6979	0.023
H10A	0.7912	0.5739	0.6962	0.032
H10B	0.8346	0.6355	0.7472	0.032
H10C	0.8799	0.6296	0.6682	0.032
H11A	0.6511	0.6019	0.6061	0.025
H11B	0.7562	0.6392	0.5720	0.025
H12A	0.6128	0.6954	0.5179	0.025
H12B	0.5456	0.7016	0.5904	0.025

Table S17. Anisotropic Displacement Parameters for **6**

Atom	u^{11}	u^{22}	u^{33}	u^{12}	u^{13}	u^{23}
Re1	0.01040(4)	0.01433(4)	0.01415(4)	-0.00048(3)	-0.00012(3)	-0.00119(3)
Cl1	0.0124(2)	0.0295(3)	0.0148(2)	-0.0024(2)	-0.00119(18)	-0.0024(2)
Cl2	0.0146(2)	0.0305(3)	0.0161(2)	0.0027(2)	0.0021(2)	-0.0034(2)
C1	0.0156(10)	0.0229(12)	0.0211(11)	-0.0035(9)	0.0031(8)	0.0000(9)
O1	0.0318(11)	0.0201(9)	0.0372(11)	-0.0074(8)	0.0032(9)	0.0032(8)
N1	0.0155(9)	0.0123(8)	0.0148(8)	-0.0002(7)	0.0018(7)	0.0005(7)
N2	0.0135(9)	0.0146(8)	0.0167(8)	0.0027(7)	-0.0001(7)	-0.0013(7)
N3	0.0148(9)	0.0176(9)	0.0177(9)	-0.0012(7)	-0.0013(7)	0.0007(7)
C2	0.0120(9)	0.0147(9)	0.0147(9)	-0.0013(8)	0.0015(7)	-0.0009(8)
C3	0.0167(10)	0.0197(10)	0.0145(10)	0.0023(9)	-0.0003(8)	0.0000(8)
C4	0.0229(12)	0.0248(12)	0.0134(10)	0.0011(10)	0.0002(9)	-0.0018(9)
C5	0.0233(12)	0.0167(11)	0.0223(11)	0.0008(9)	0.0048(10)	-0.0046(9)
C6	0.0173(11)	0.0141(10)	0.0243(12)	0.0013(8)	0.0018(9)	0.0023(9)
C7	0.0139(10)	0.0174(10)	0.0159(10)	-0.0002(8)	-0.0007(8)	0.0011(8)
F3	0.0246(8)	0.0233(7)	0.0165(7)	0.0062(6)	-0.0033(6)	0.0022(6)
F4	0.0426(10)	0.0319(9)	0.0149(7)	0.0042(8)	-0.0033(7)	-0.0056(6)
F5	0.0388(10)	0.0222(8)	0.0310(9)	0.0052(7)	0.0067(7)	-0.0107(7)
F6	0.0293(8)	0.0181(7)	0.0308(9)	0.0075(6)	0.0009(7)	0.0040(6)
F7	0.0235(7)	0.0207(7)	0.0172(7)	0.0028(6)	-0.0038(6)	0.0023(6)
C8	0.0161(10)	0.0160(10)	0.0174(10)	-0.0007(8)	0.0018(8)	0.0025(8)
C9	0.0163(11)	0.0168(10)	0.0232(11)	-0.0026(9)	0.0007(9)	-0.0002(9)
C10	0.0208(12)	0.0182(11)	0.0253(12)	0.0067(9)	-0.0041(10)	0.0003(9)
C11	0.0240(12)	0.0180(11)	0.0202(11)	-0.0001(10)	-0.0038(9)	-0.0037(9)
C12	0.0245(12)	0.0178(11)	0.0209(11)	-0.0036(9)	-0.0065(10)	0.0000(9)
C13	0.0189(11)	0.0178(10)	0.0133(10)	-0.0006(9)	-0.0039(8)	0.0008(8)
C14	0.0183(11)	0.0212(11)	0.0150(10)	-0.0022(9)	-0.0024(8)	-0.0012(9)
C15	0.0223(12)	0.0208(11)	0.0225(12)	0.0017(10)	-0.0112(10)	-0.0035(9)
C16	0.0372(15)	0.0164(11)	0.0192(11)	-0.0039(11)	-0.0100(11)	0.0011(9)
C17	0.0346(15)	0.0268(13)	0.0163(11)	-0.0102(11)	-0.0012(10)	0.0044(10)
C18	0.0213(11)	0.0238(12)	0.0185(11)	-0.0036(10)	-0.0015(9)	-0.0006(9)
F14	0.0176(7)	0.0339(9)	0.0198(7)	-0.0017(6)	0.0013(6)	0.0011(6)
F15	0.0252(8)	0.0296(9)	0.0374(9)	0.0078(7)	-0.0130(7)	-0.0028(7)
F16	0.0545(12)	0.0273(9)	0.0296(9)	-0.0033(8)	-0.0201(9)	0.0108(7)
F17	0.0453(12)	0.0472(12)	0.0230(8)	-0.0163(9)	0.0013(8)	0.0145(8)
F18	0.0224(8)	0.0431(10)	0.0274(8)	0.0016(7)	0.0070(7)	0.0050(8)

Table S18. Bond Lengths for **6**

Re1-C1	1.862(3)	C8-C9	1.507(3)
Re1-N3	1.920(2)	C8-H8A	0.9900
Re1-N1	2.200(2)	C8-H8B	0.9900
Re1-N2	2.262(2)	C9-H9A	0.9900
Re1-Cl2	2.3685(6)	C9-H9B	0.9900
Re1-Cl1	2.4361(6)	C10-H10A	0.9800
C1-O1	1.168(3)	C10-H10B	0.9800
N1-C2	1.448(3)	C10-H10C	0.9800
N1-C8	1.505(3)	C11-C12	1.515(4)
N2-C11	1.485(3)	C11-H11A	0.9900
N2-C10	1.489(3)	C11-H11B	0.9900
N2-C9	1.494(3)	C12-H12A	0.9900
N3-C13	1.426(3)	C12-H12B	0.9900
N3-C12	1.491(3)	C13-C14	1.391(3)
C2-C3	1.389(3)	C13-C18	1.394(4)
C2-C7	1.393(3)	C14-F14	1.337(3)
C3-F3	1.338(3)	C14-C15	1.381(3)
C3-C4	1.395(3)	C15-F15	1.342(3)
C4-F4	1.343(3)	C15-C16	1.378(4)
C4-C5	1.367(4)	C16-F16	1.339(3)
C5-F5	1.334(3)	C16-C17	1.368(4)
C5-C6	1.381(4)	C17-F17	1.342(3)
C6-F6	1.337(3)	C17-C18	1.383(4)
C6-C7	1.379(3)	C18-F18	1.335(3)
C7-F7	1.335(3)		

Table S19. Bond Angles for **6**

C1-Re1-N3	96.74(10)	N1-C8-H8A	110.4
C1-Re1-N1	98.15(9)	C9-C8-H8A	110.4
N3-Re1-N1	93.97(8)	N1-C8-H8B	110.4
C1-Re1-N2	174.44(9)	C9-C8-H8B	110.4
N3-Re1-N2	79.47(8)	H8A-C8-H8B	108.6
N1-Re1-N2	78.18(7)	N2-C9-C8	110.39(19)
C1-Re1-Cl2	93.00(8)	N2-C9-H9A	109.6
N3-Re1-Cl2	96.37(6)	C8-C9-H9A	109.6
N1-Re1-Cl2	163.79(5)	N2-C9-H9B	109.6
N2-Re1-Cl2	91.46(5)	C8-C9-H9B	109.6
C1-Re1-Cl1	91.53(8)	H9A-C9-H9B	108.1
N3-Re1-Cl1	170.40(6)	N2-C10-H10A	109.5
N1-Re1-Cl1	80.01(6)	N2-C10-H10B	109.5
N2-Re1-Cl1	91.93(5)	H10A-C10-H10B	109.5
Cl2-Re1-Cl1	88.01(2)	N2-C10-H10C	109.5
O1-C1-Re1	179.4(2)	H10A-C10-H10C	109.5
C2-N1-C8	115.12(18)	H10B-C10-H10C	109.5
C2-N1-Re1	121.23(14)	N2-C11-C12	109.5(2)
C8-N1-Re1	107.95(14)	N2-C11-H11A	109.8
C11-N2-C10	108.67(19)	C12-C11-H11A	109.8
C11-N2-C9	109.93(19)	N2-C11-H11B	109.8
C10-N2-C9	108.94(19)	C12-C11-H11B	109.8
C11-N2-Re1	104.36(14)	H11A-C11-H11B	108.2
C10-N2-Re1	115.99(15)	N3-C12-C11	108.1(2)
C9-N2-Re1	108.80(14)	N3-C12-H12A	110.1
C13-N3-C12	111.73(19)	C11-C12-H12A	110.1
C13-N3-Re1	127.82(16)	N3-C12-H12B	110.1
C12-N3-Re1	120.44(16)	C11-C12-H12B	110.1
C3-C2-C7	116.7(2)	H12A-C12-H12B	108.4
C3-C2-N1	126.5(2)	C14-C13-C18	117.0(2)
C7-C2-N1	116.5(2)	C14-C13-N3	121.3(2)
F3-C3-C2	122.9(2)	C18-C13-N3	121.6(2)
F3-C3-C4	116.4(2)	F14-C14-C15	118.6(2)
C2-C3-C4	120.7(2)	F14-C14-C13	119.5(2)
F4-C4-C5	119.5(2)	C15-C14-C13	121.9(2)
F4-C4-C3	119.2(2)	F15-C15-C16	120.5(2)
C5-C4-C3	121.3(2)	F15-C15-C14	119.9(2)
F5-C5-C4	121.1(2)	C16-C15-C14	119.6(2)
F5-C5-C6	120.0(2)	F16-C16-C17	120.5(3)
C4-C5-C6	118.9(2)	F16-C16-C15	119.5(3)
F6-C6-C7	119.8(2)	C17-C16-C15	120.0(2)
F6-C6-C5	120.3(2)	F17-C17-C16	119.7(2)
C7-C6-C5	119.9(2)	F17-C17-C18	120.0(3)
F7-C7-C6	118.7(2)	C16-C17-C18	120.3(2)
F7-C7-C2	118.9(2)	F18-C18-C17	118.6(2)
C6-C7-C2	122.4(2)	F18-C18-C13	120.1(2)
N1-C8-C9	106.46(19)	C17-C18-C13	121.2(3)

Table S20. Torsion Angles for **6**

N3-Re1-C1-O1	119(30)	F4-C4-C5-C6	179.7(2)
N1-Re1-C1-O1	24(30)	C3-C4-C5-C6	-2.1(4)
N2-Re1-C1-O1	72(30)	F5-C5-C6-F6	-0.2(4)
Cl2-Re1-C1-O1	-144(30)	C4-C5-C6-F6	179.9(2)
Cl1-Re1-C1-O1	-56(30)	F5-C5-C6-C7	-179.0(2)
C1-Re1-N1-C2	-22.89(19)	C4-C5-C6-C7	1.2(4)
N3-Re1-N1-C2	-120.28(17)	F6-C6-C7-F7	0.1(3)
N2-Re1-N1-C2	161.35(18)	C5-C6-C7-F7	178.8(2)
Cl2-Re1-N1-C2	110.2(2)	F6-C6-C7-C2	-177.7(2)
Cl1-Re1-N1-C2	67.24(16)	C5-C6-C7-C2	1.0(4)
C1-Re1-N1-C8	-158.79(16)	C3-C2-C7-F7	-180.0(2)
N3-Re1-N1-C8	103.81(15)	N1-C2-C7-F7	-4.9(3)
N2-Re1-N1-C8	25.45(14)	C3-C2-C7-C6	-2.2(3)
Cl2-Re1-N1-C8	-25.8(3)	N1-C2-C7-C6	172.9(2)
Cl1-Re1-N1-C8	-68.67(14)	C2-N1-C8-C9	169.50(19)
C1-Re1-N2-C11	72.8(10)	Re1-N1-C8-C9	-51.6(2)
N3-Re1-N2-C11	25.55(15)	C11-N2-C9-C8	-148.3(2)
N1-Re1-N2-C11	121.91(15)	C10-N2-C9-C8	92.7(2)
Cl2-Re1-N2-C11	-70.66(14)	Re1-N2-C9-C8	-34.6(2)
Cl1-Re1-N2-C11	-158.72(14)	N1-C8-C9-N2	57.9(2)
C1-Re1-N2-C10	-167.7(9)	C10-N2-C11-C12	-167.9(2)
N3-Re1-N2-C10	145.05(18)	C9-N2-C11-C12	73.0(3)
N1-Re1-N2-C10	-118.59(17)	Re1-N2-C11-C12	-43.5(2)
Cl2-Re1-N2-C10	48.84(16)	C13-N3-C12-C11	159.2(2)
Cl1-Re1-N2-C10	-39.22(16)	Re1-N3-C12-C11	-21.2(3)
C1-Re1-N2-C9	-44.5(10)	N2-C11-C12-N3	43.5(3)
N3-Re1-N2-C9	-91.76(16)	C12-N3-C13-C14	71.6(3)
N1-Re1-N2-C9	4.60(15)	Re1-N3-C13-C14	-108.0(2)
Cl2-Re1-N2-C9	172.03(14)	C12-N3-C13-C18	-105.6(3)
Cl1-Re1-N2-C9	83.98(14)	Re1-N3-C13-C18	74.9(3)
C1-Re1-N3-C13	1.2(2)	C18-C13-C14-F14	176.4(2)
N1-Re1-N3-C13	99.9(2)	N3-C13-C14-F14	-0.9(3)
N2-Re1-N3-C13	177.1(2)	C18-C13-C14-C15	-2.5(4)
Cl2-Re1-N3-C13	-92.6(2)	N3-C13-C14-C15	-179.8(2)
Cl1-Re1-N3-C13	150.6(3)	F14-C14-C15-F15	2.1(3)
C1-Re1-N3-C12	-178.32(19)	C13-C14-C15-F15	-179.0(2)
N1-Re1-N3-C12	-79.61(19)	F14-C14-C15-C16	-177.0(2)
N2-Re1-N3-C12	-2.43(18)	C13-C14-C15-C16	1.9(4)
Cl2-Re1-N3-C12	87.88(18)	F15-C15-C16-F16	0.2(4)
Cl1-Re1-N3-C12	-28.9(5)	C14-C15-C16-F16	179.3(2)
C8-N1-C2-C3	25.3(3)	F15-C15-C16-C17	-179.1(2)
Re1-N1-C2-C3	-107.7(2)	C14-C15-C16-C17	-0.1(4)
C8-N1-C2-C7	-149.1(2)	F16-C16-C17-F17	-1.3(4)
Re1-N1-C2-C7	77.8(2)	C15-C16-C17-F17	178.0(2)

C7-C2-C3-F3	179.2(2)	F16-C16-C17-C18	179.5(2)
N1-C2-C3-F3	4.7(4)	C15-C16-C17-C18	-1.1(4)
C7-C2-C3-C4	1.2(4)	F17-C17-C18-F18	-0.8(4)
N1-C2-C3-C4	-173.2(2)	C16-C17-C18-F18	178.4(2)
F3-C3-C4-F4	0.9(4)	F17-C17-C18-C13	-178.7(2)
C2-C3-C4-F4	179.0(2)	C16-C17-C18-C13	0.5(4)
F3-C3-C4-C5	-177.2(2)	C14-C13-C18-F18	-176.5(2)
C2-C3-C4-C5	0.9(4)	N3-C13-C18-F18	0.7(4)
F4-C4-C5-F5	-0.1(4)	C14-C13-C18-C17	1.3(4)
C3-C4-C5-F5	178.0(2)	N3-C13-C18-C17	178.6(2)

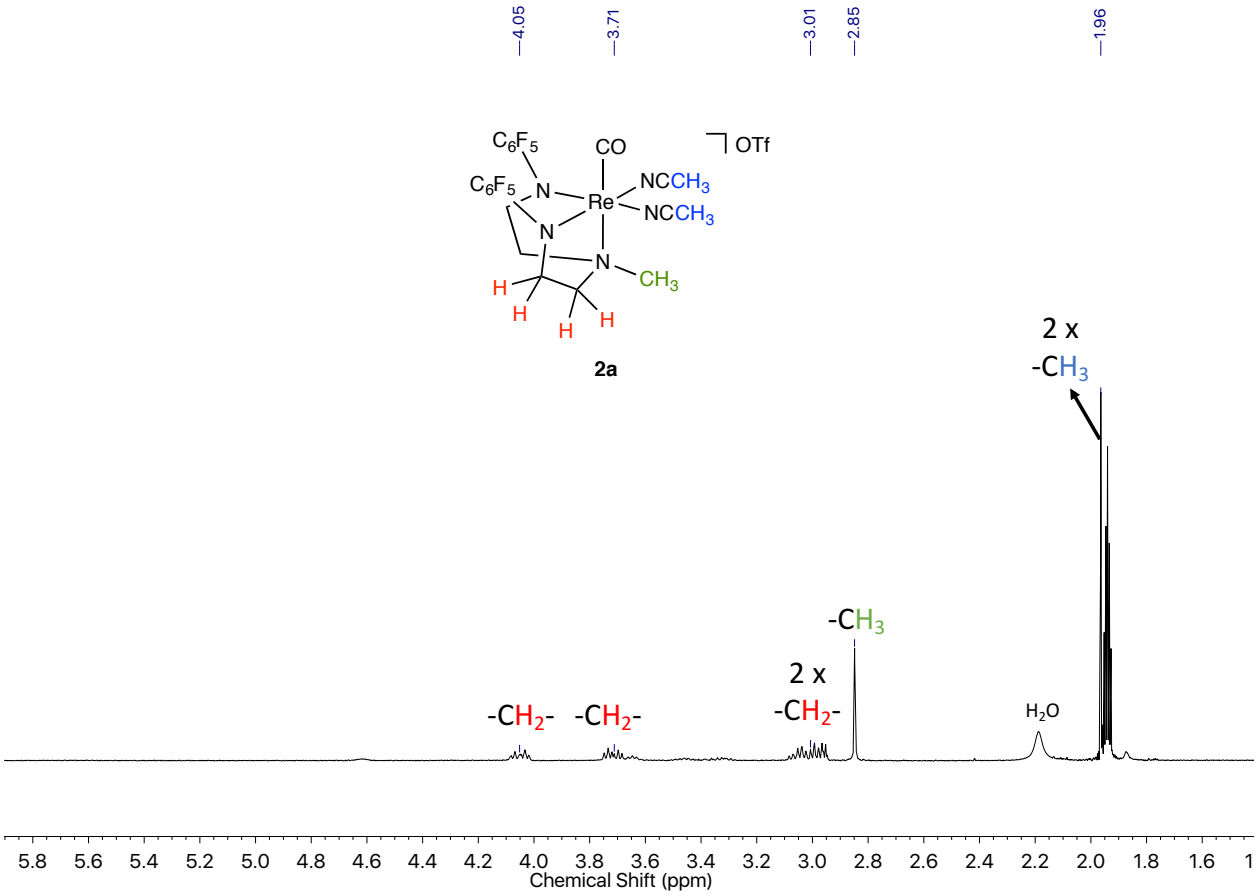


Figure S14. ^{1}H NMR (376 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)($NCCH_3$)$_2$][OTf] (**2a**).

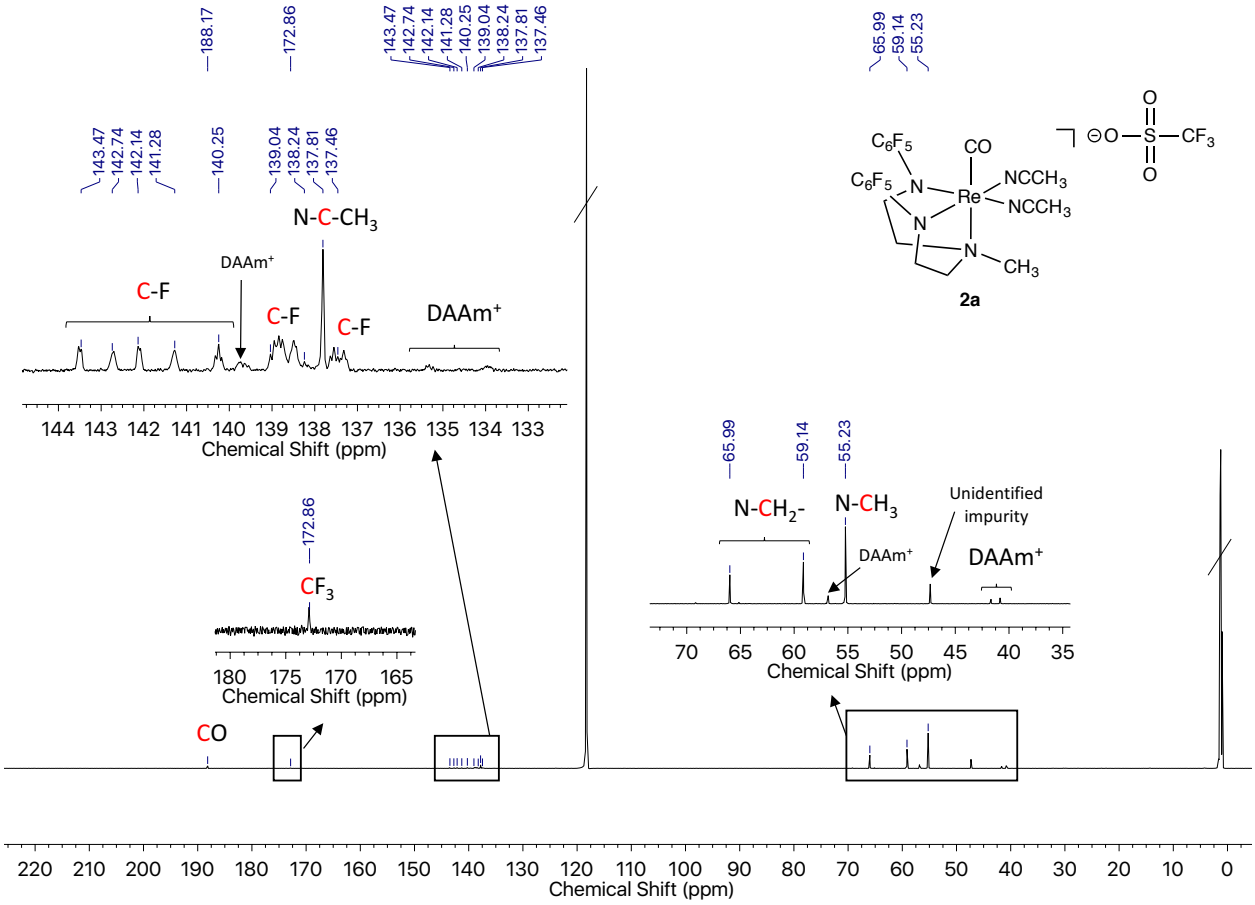


Figure S15. ^{13}C NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}C_6F_5)Re(CO)(NCCH_3)_2][OTf]$ (**2a**). Residual $[DAAm\text{-}C_6F_5]^+$ and impurity peaks observed at 139.71 ppm, 56.83 ppm, 41.68 ppm and 40.83 ppm, and 47.53 respectively.

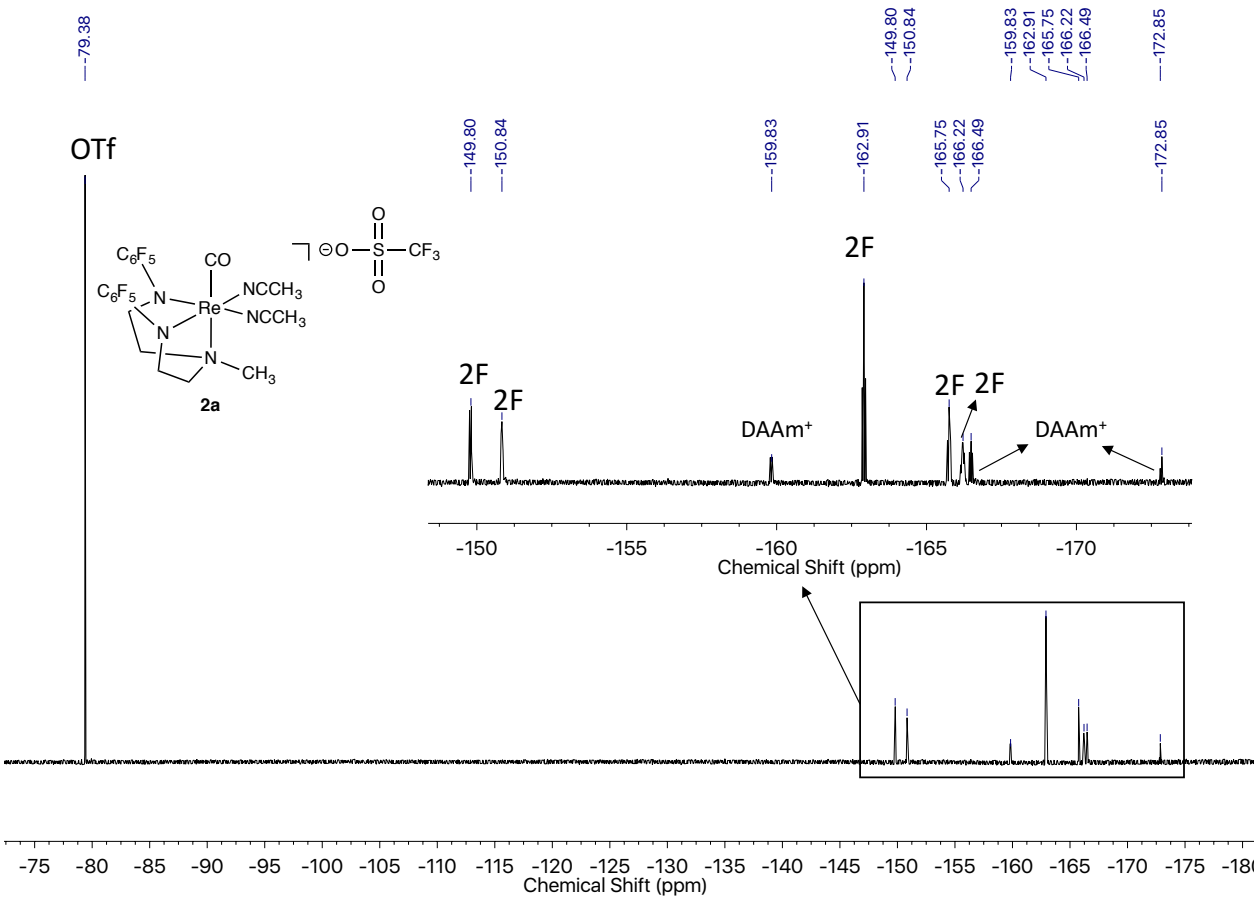


Figure S16. ^{19}F NMR (376 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)($NCCH_3$)$_2$][OTf] (**2a**). Residual [DAAm-C_6F_5]$^+$ peaks observed at -159.83 ppm, -166.49 ppm and -172.85 ppm.

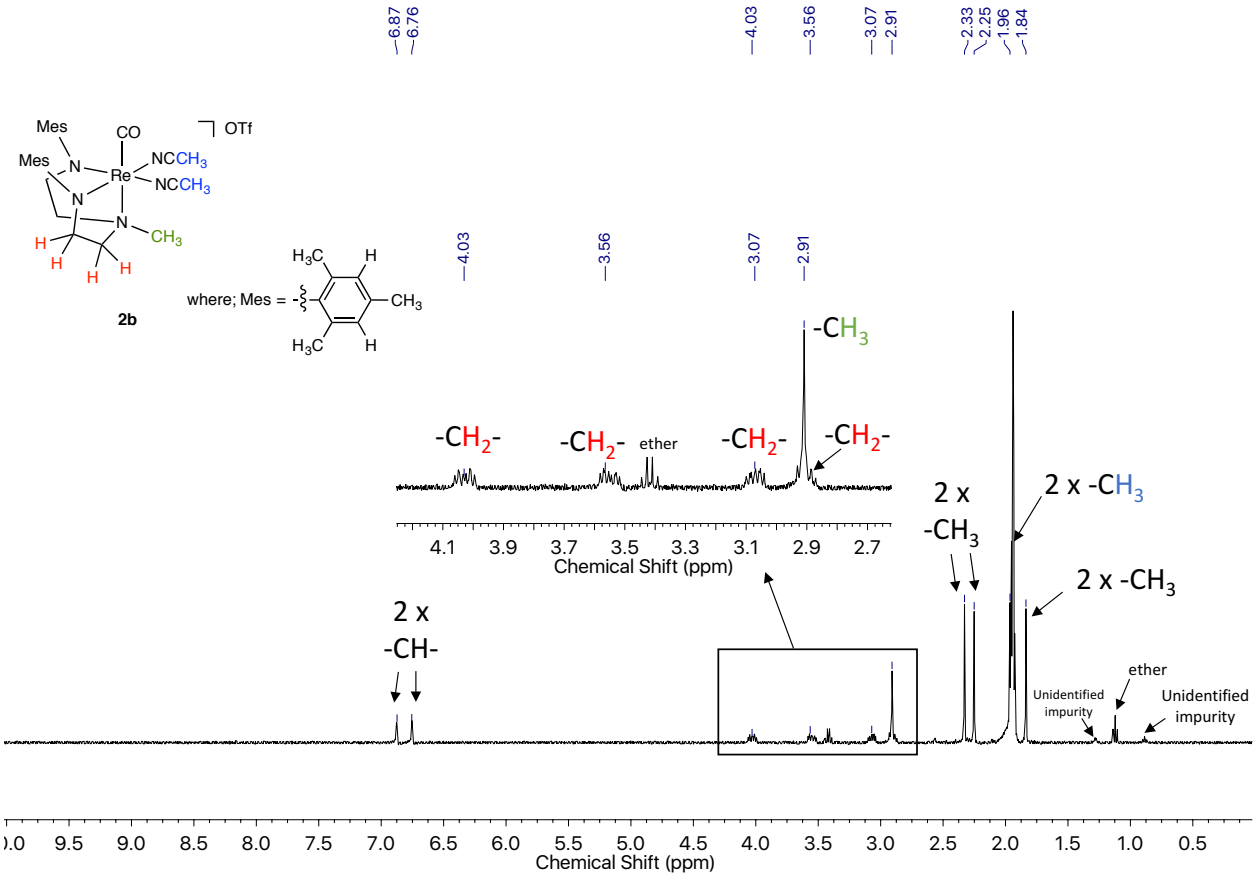


Figure S17. ^{1}H NMR (400 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][OTf] (**2b**). Residual ether and unidentified impurity peaks observed at 3.40 ppm and 1.12 ppm, and 1.27 ppm and 0.88 ppm respectively.

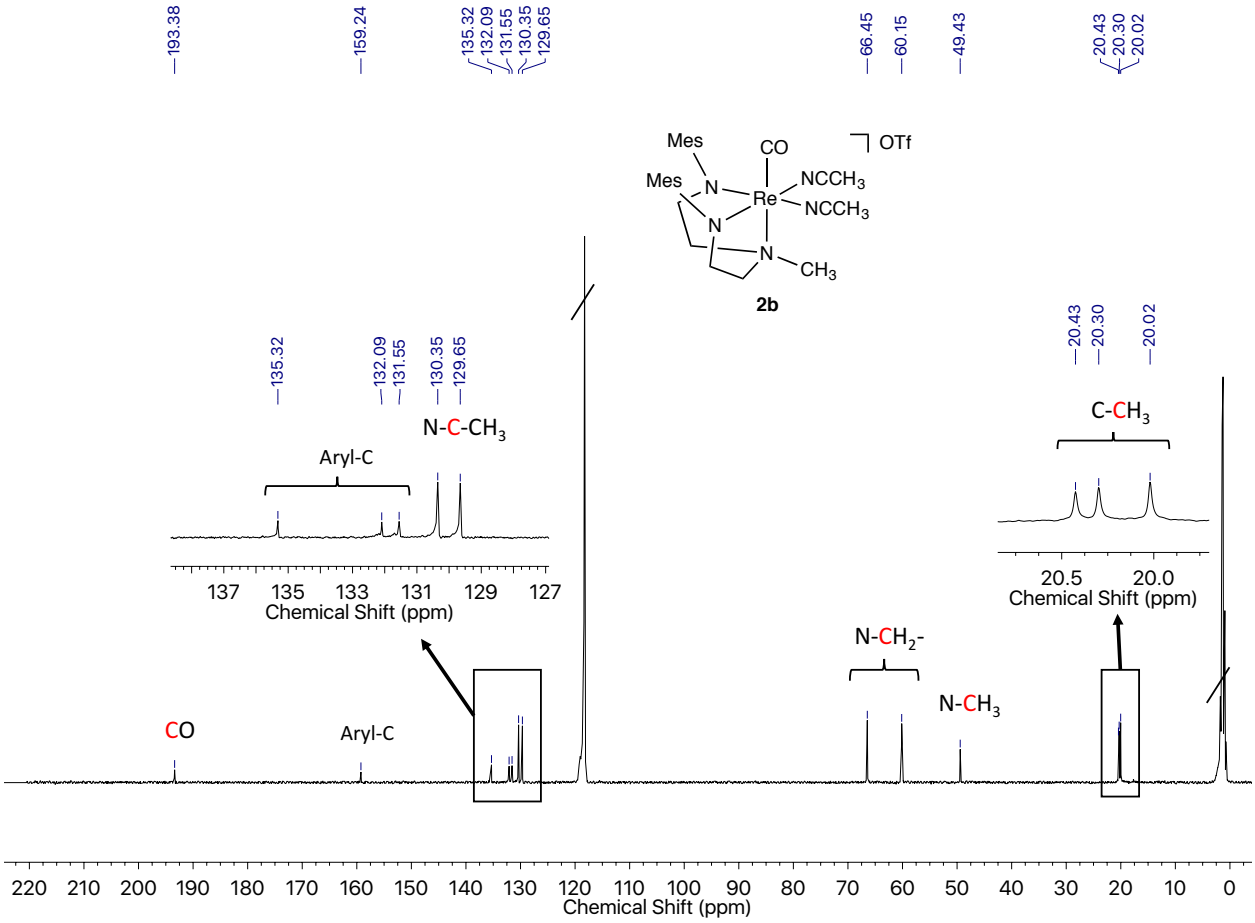


Figure S18. ^{13}C NMR (126 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][OTf] (**2b**).

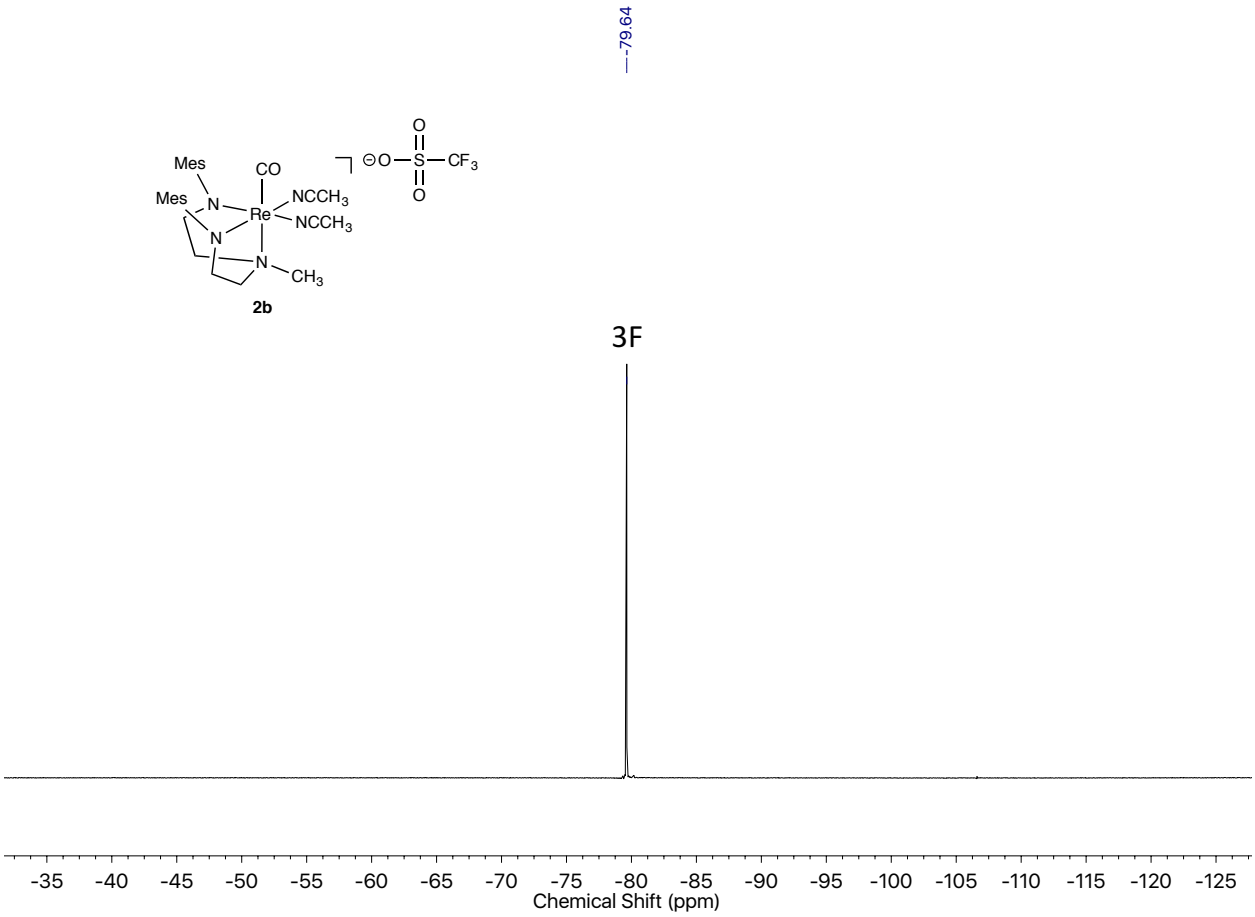


Figure S19. ^{19}F NMR (376 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][OTf] (**2b**).

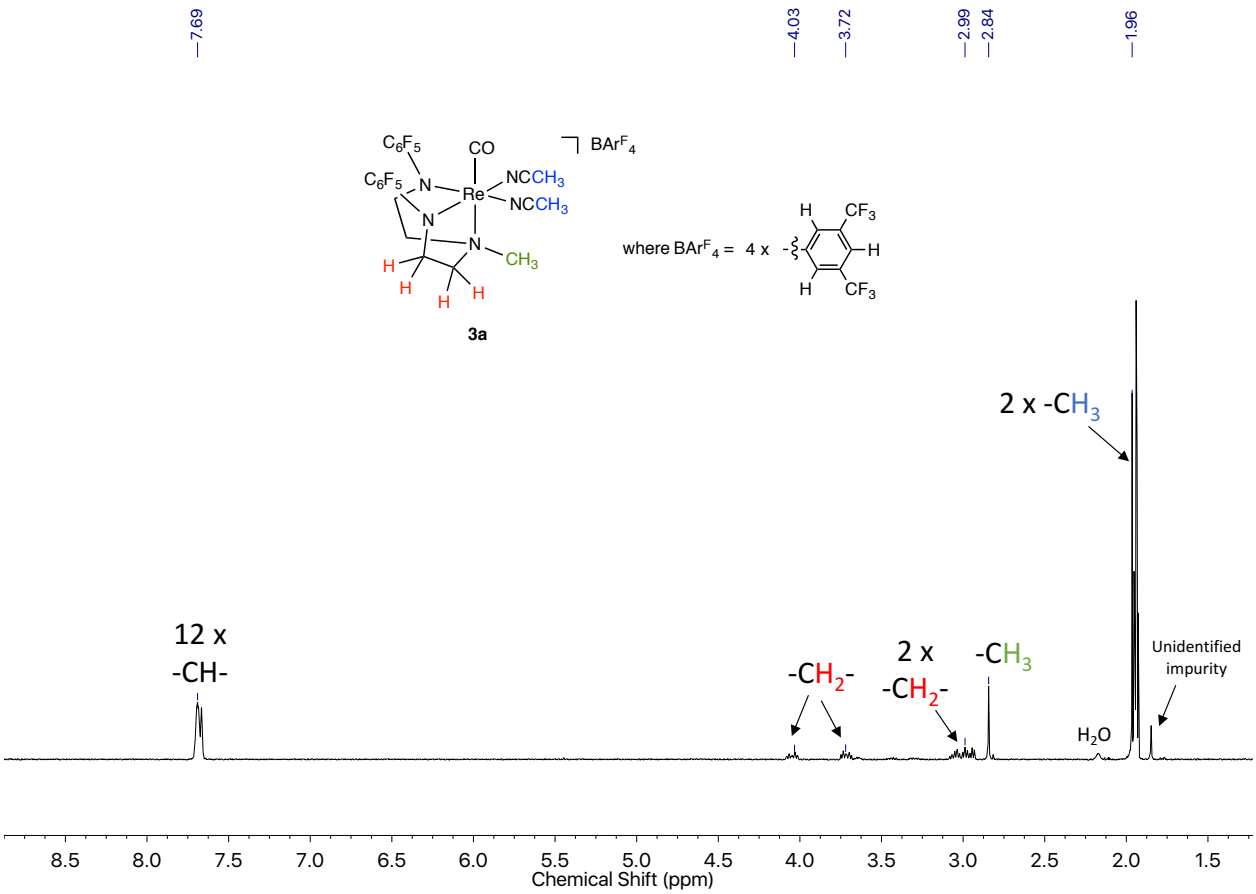


Figure S20. ^{1}H NMR (376 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)($NCCH_3$)$_2$][BAr^F_4] (**3a**). Residual H_2O and unidentified impurity peaks observed at 2.17 ppm and 1.85 ppm respectively.

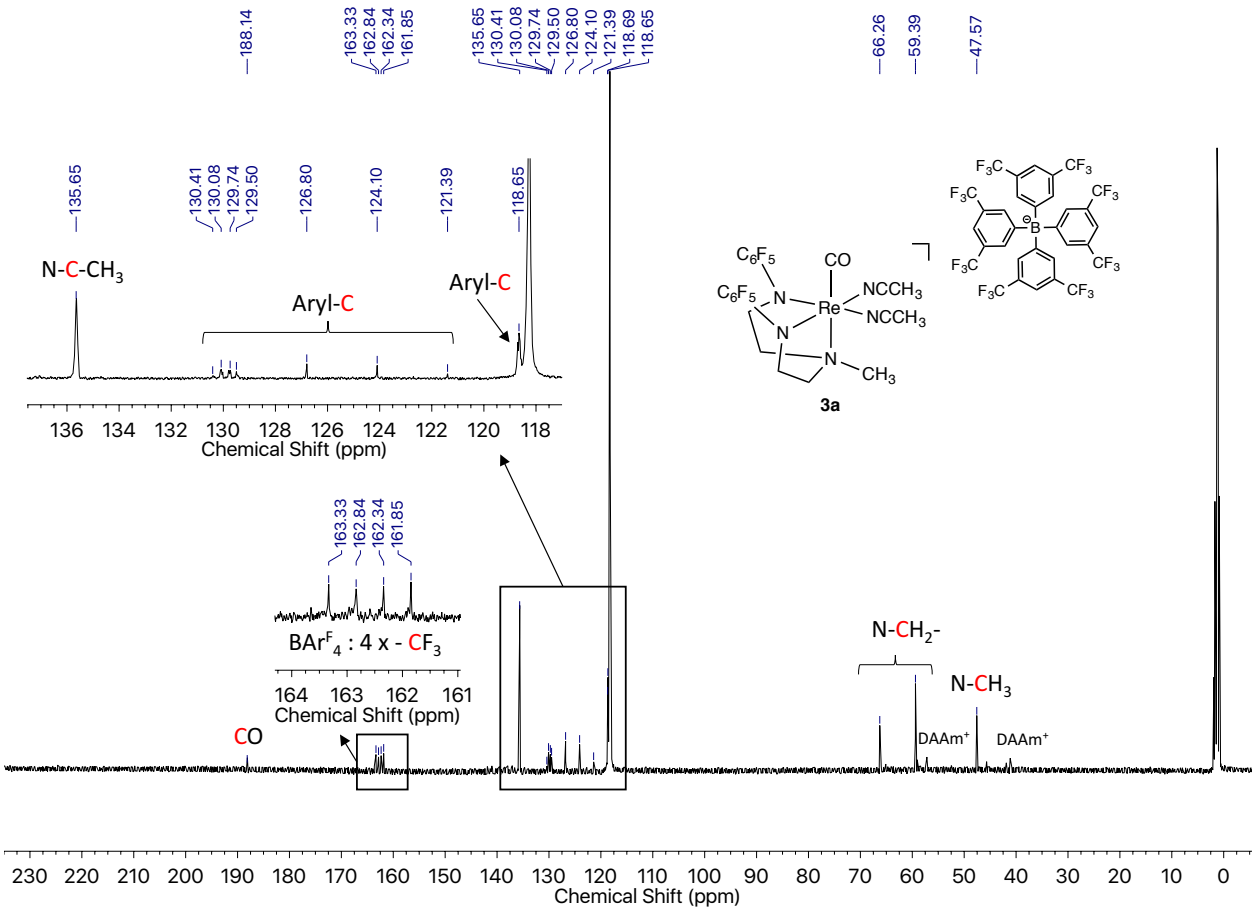


Figure S21. ^{13}C NMR (101 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)$(NCCH_3)_2$][BAr^F_4] (**3a**). Residual [DAAm-C_6F_5]$^+$ peaks observed at 57.18 ppm, 41.88 ppm and 41.10 ppm.

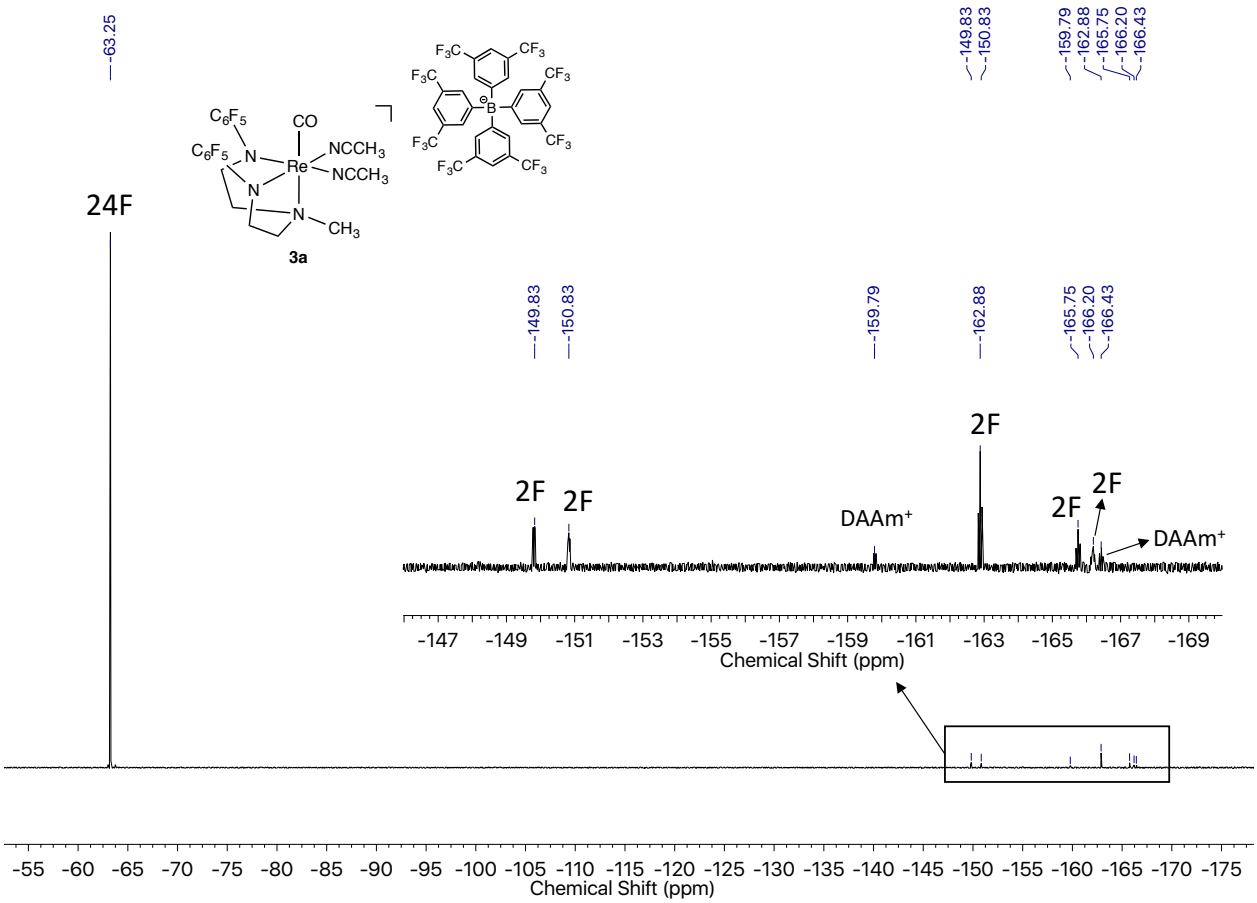


Figure S22. ^{19}F NMR (376 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)($NCCH_3$)$_2$][$BAr^F{}_4$] (**3a**). Residual [DAAm-C_6F_5]$^+$ peaks observed at -159.83 ppm, -166.49 ppm and -172.85 ppm.

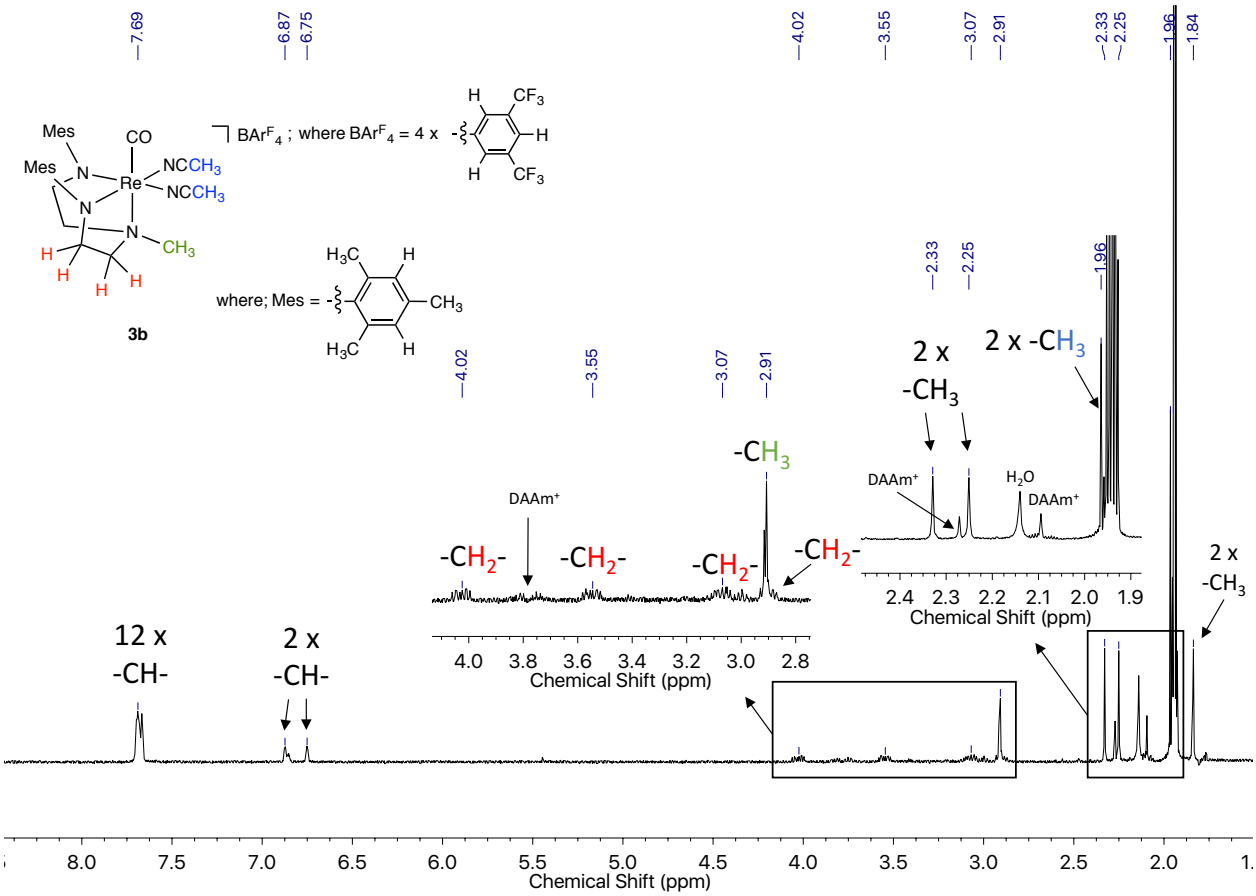


Figure S23. ^{1}H NMR (376 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)$(NCCH_3)_2$][BAr^F_4] (**3b**). Residual [DAAm-Mes]$^+$ and H_2O peaks observed at 3.79 ppm, 2.27 ppm, 2.09 ppm and 2.14 ppm respectively.

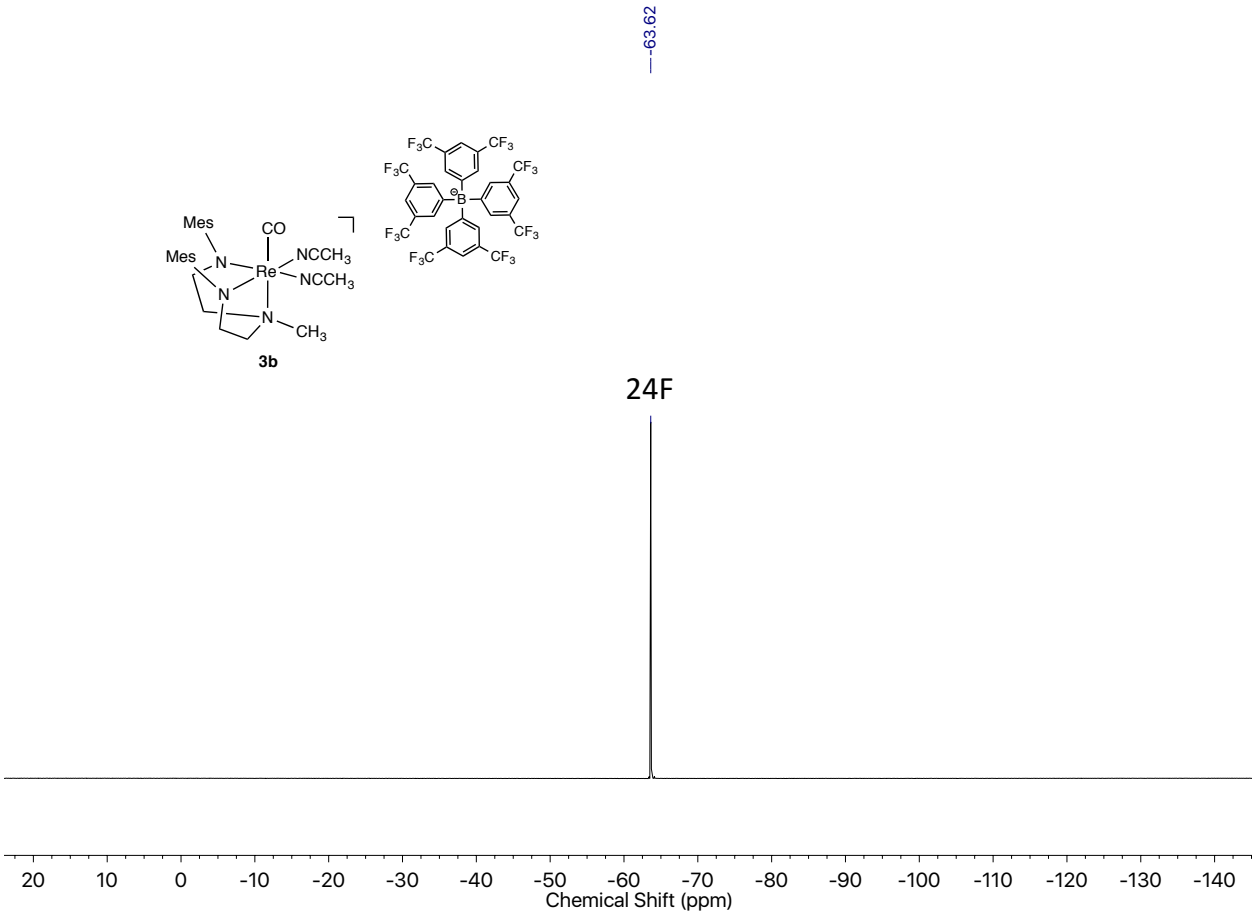


Figure S24. ^{19}F NMR (376 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][BAr^F_4] (**3b**).

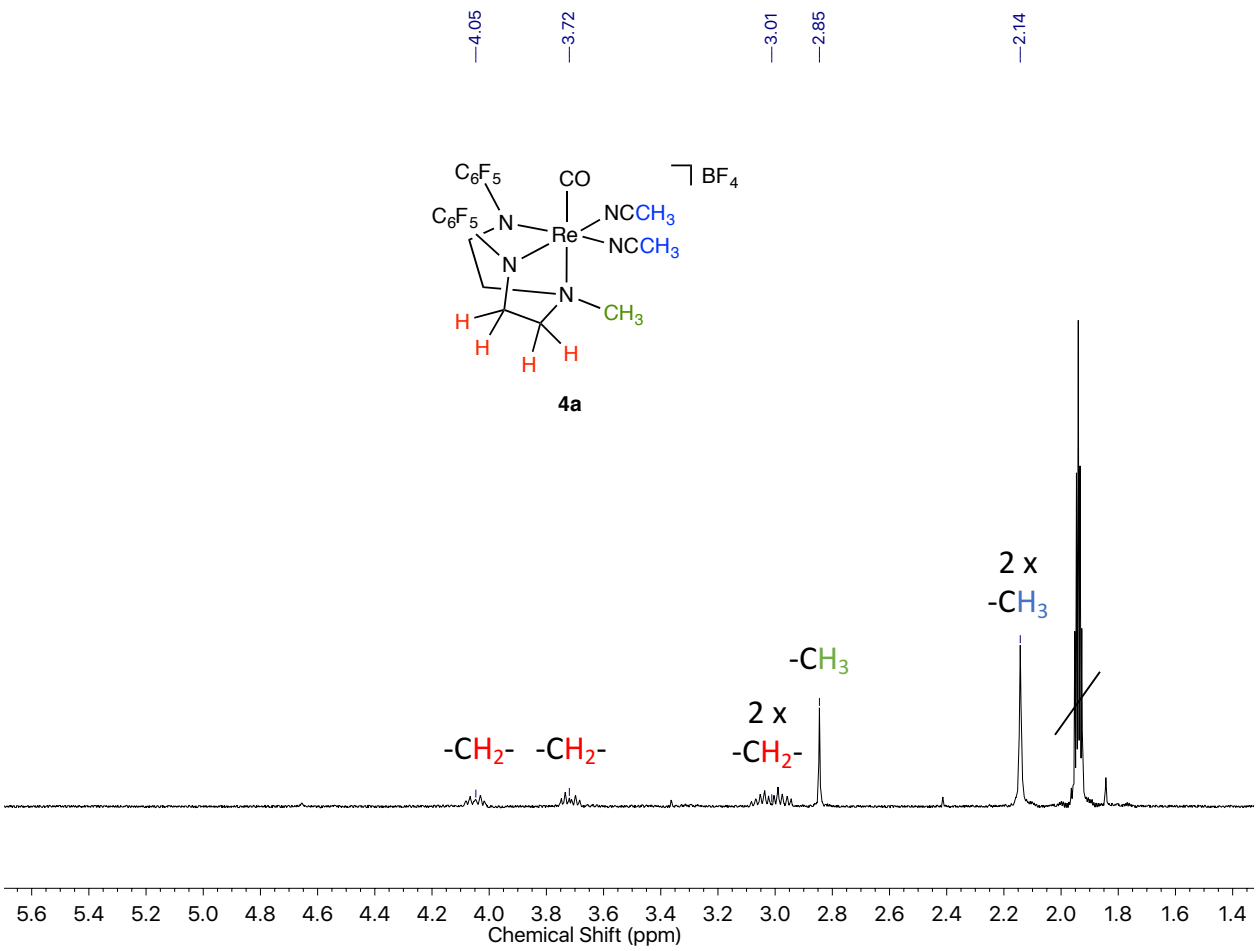


Figure S25. ^{1}H NMR (176 MHz, CD_3CN) spectrum of [(DAAm-C_6F_5)Re(CO)($NCCH_3$)$_2$][BF_4] (**4a**).

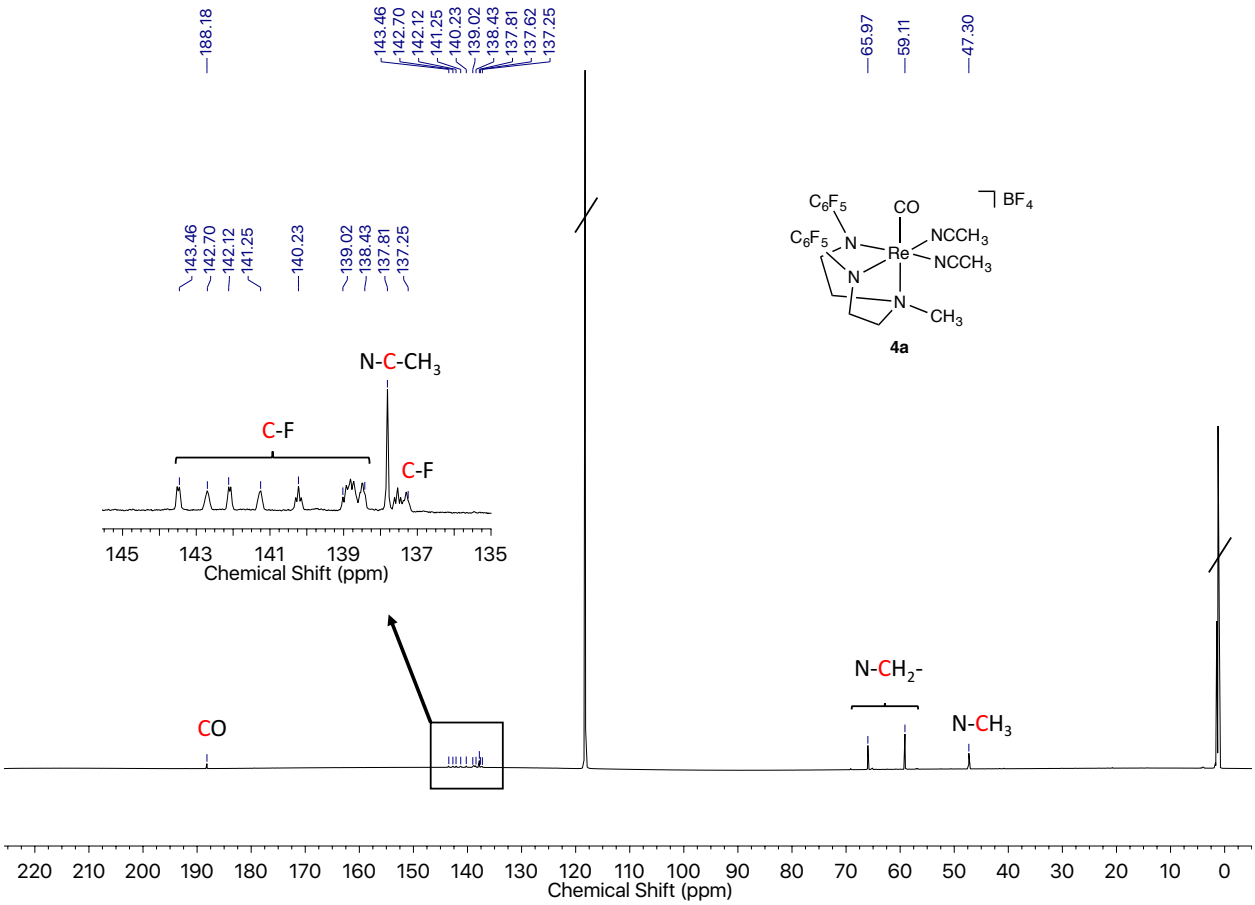
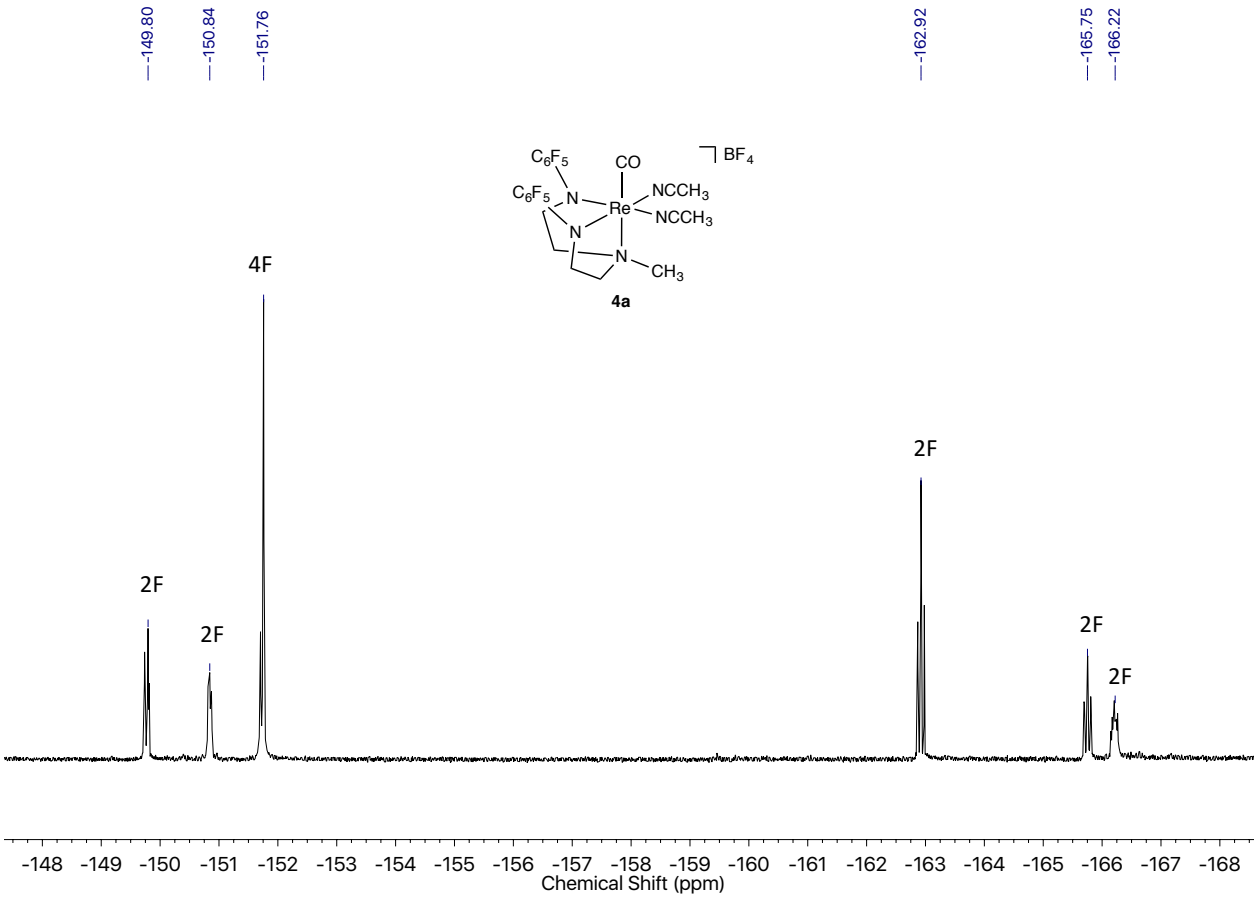


Figure S26. ^{13}C NMR (176 MHz, CD_3CN) spectrum of $[(DAAm\text{-}C_6F_5)Re(CO)(NCCH_3)_2][BF_4]$ (**4a**).



4a

Figure S27. ^{19}F NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}C_6F_5)Re(CO)(NCCH_3)_2][BF_4]$ (**4a**).

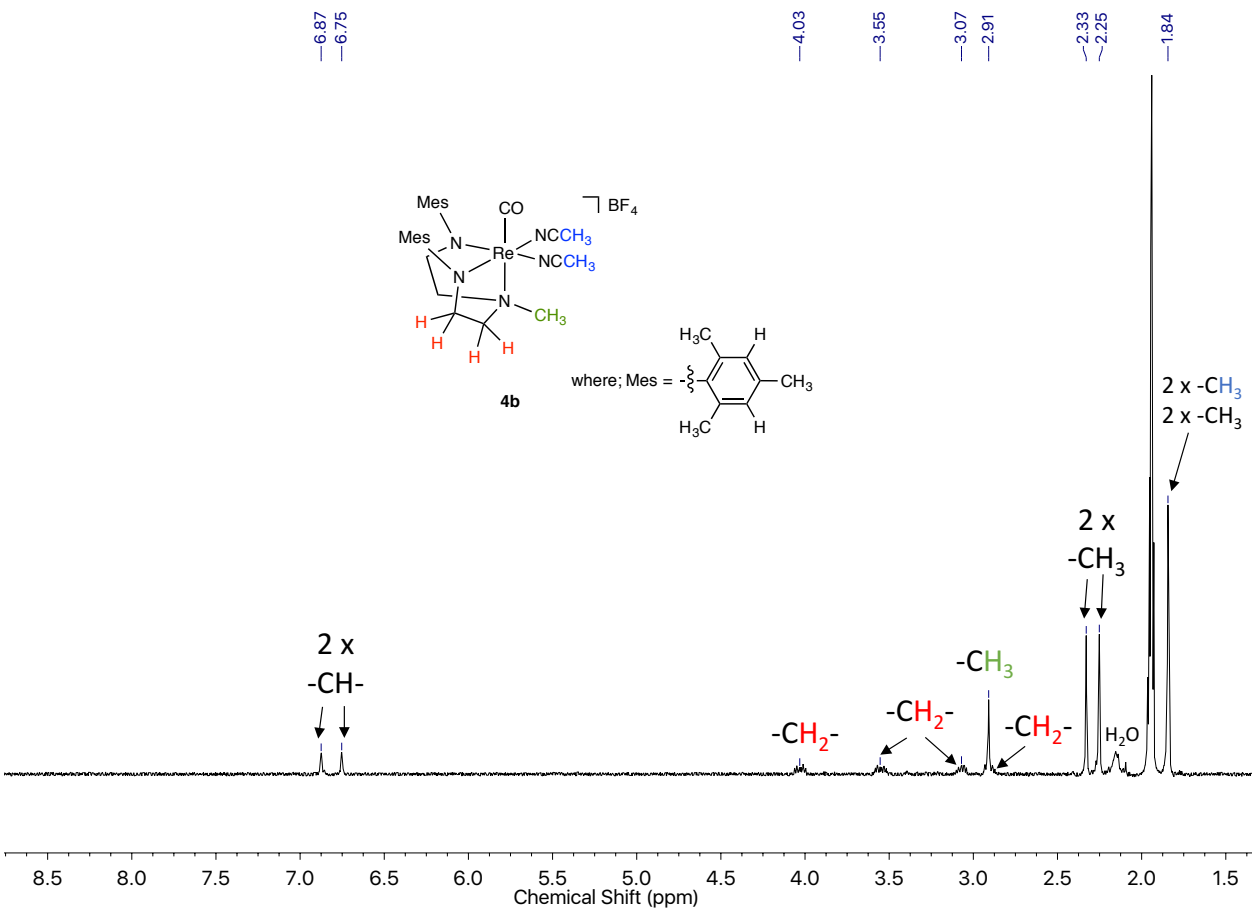


Figure S28. ^{1}H NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][BF_4]$ (**4b**).

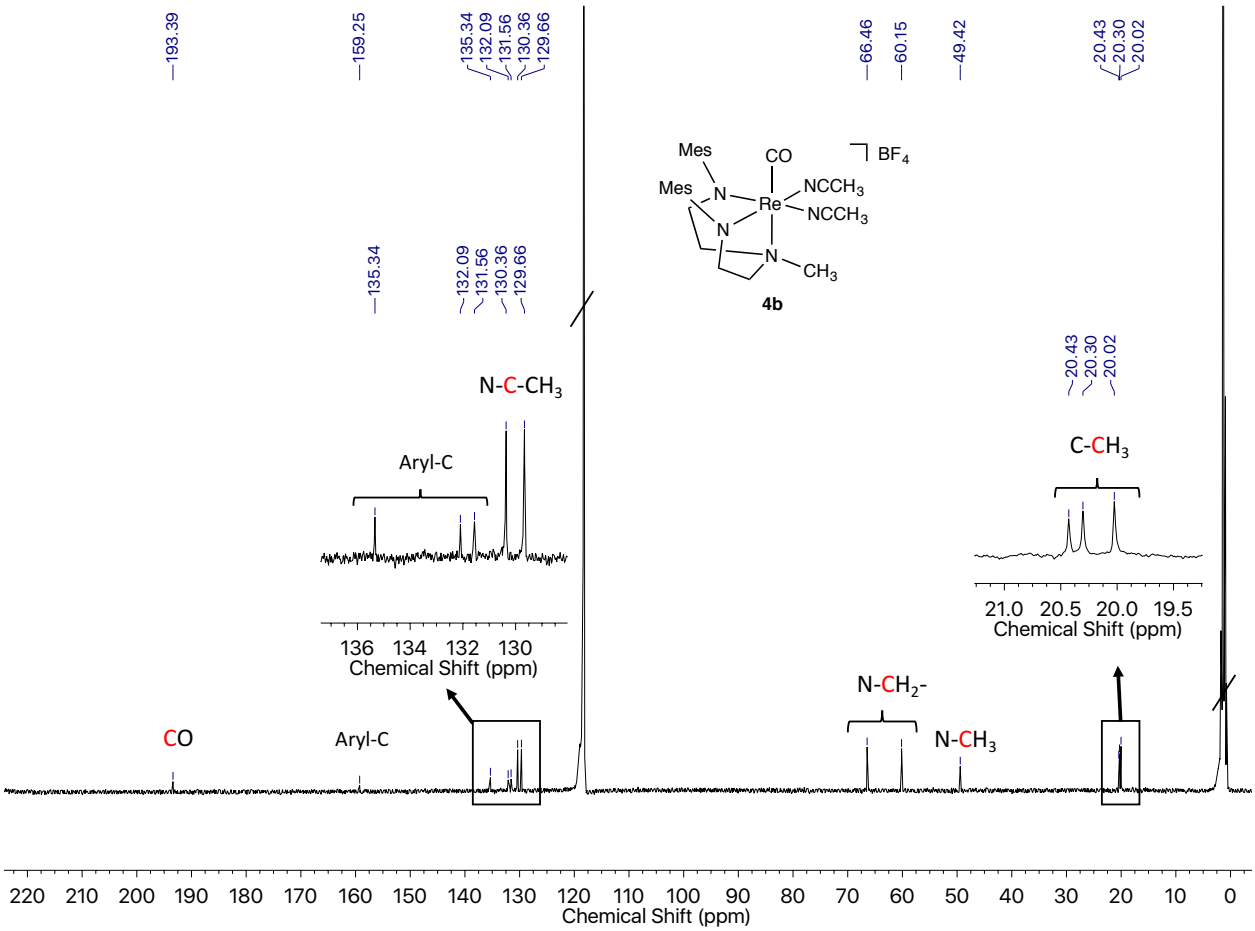


Figure S29. ^{13}C NMR (376 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][BF_4] (**4b**).

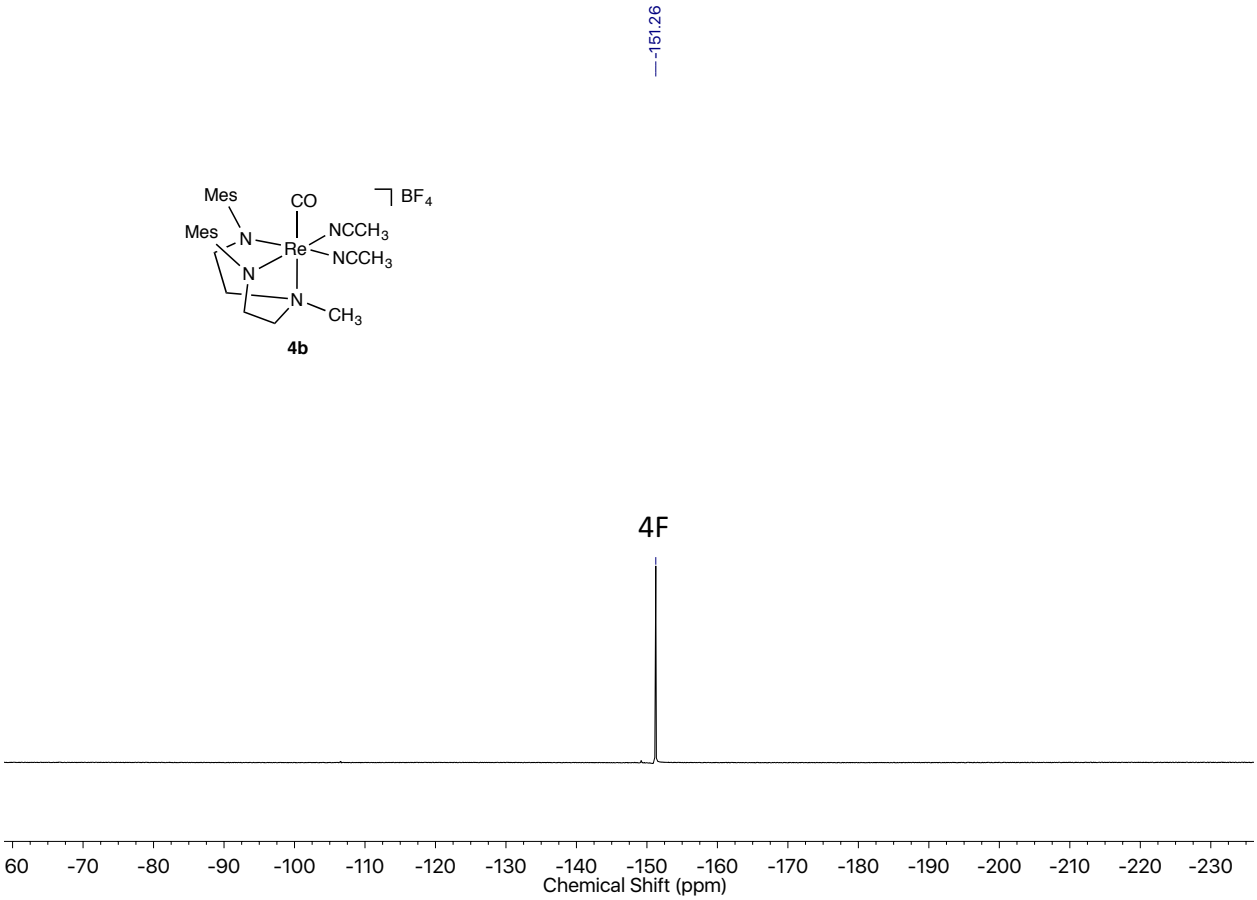


Figure S30. ^{19}F NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][BF_4]$ (**4b**).

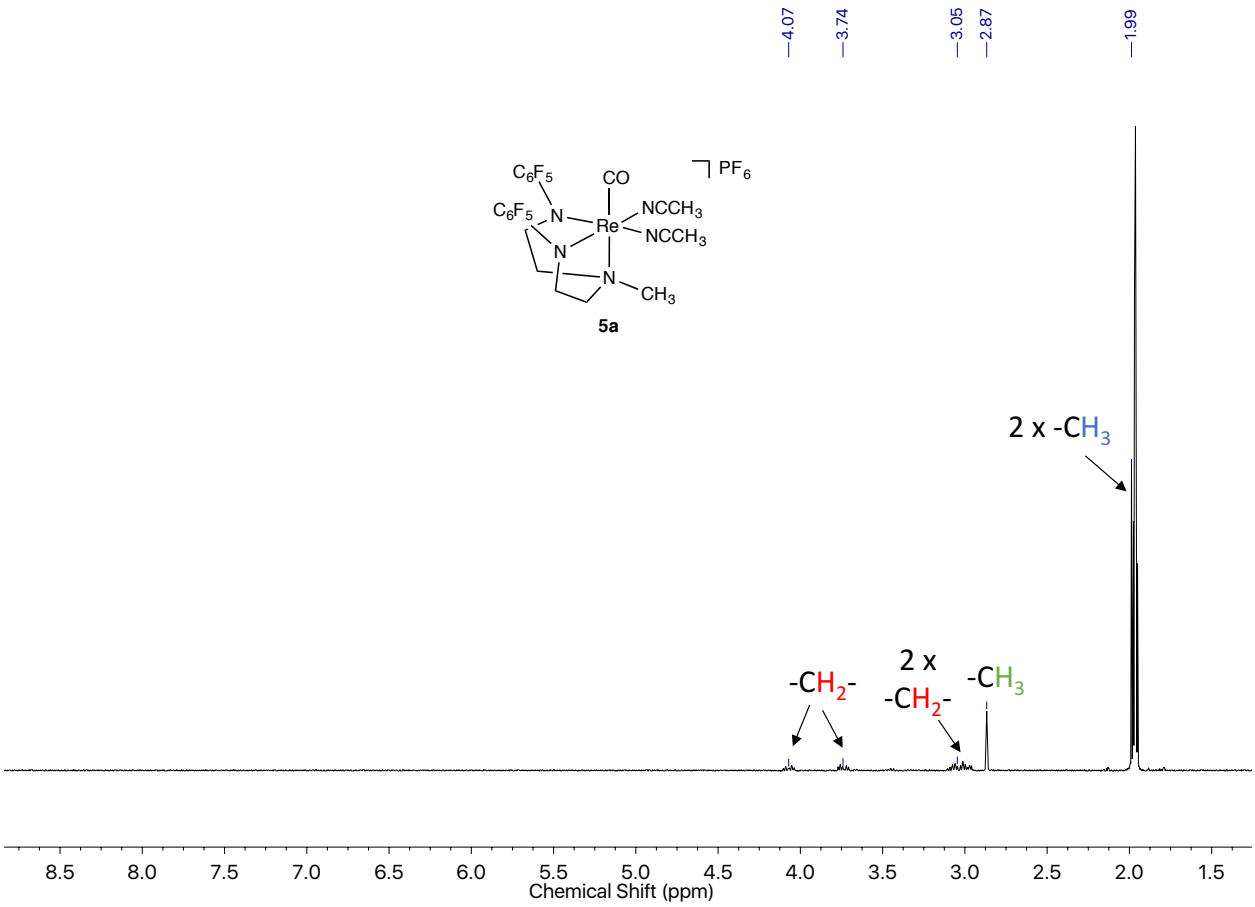


Figure S31. ^{1}H NMR (376 MHz, CD_3CN) spectrum of [(DAAm-Mes)Re(CO)($NCCH_3$)$_2$][PF_6] (**5a**).

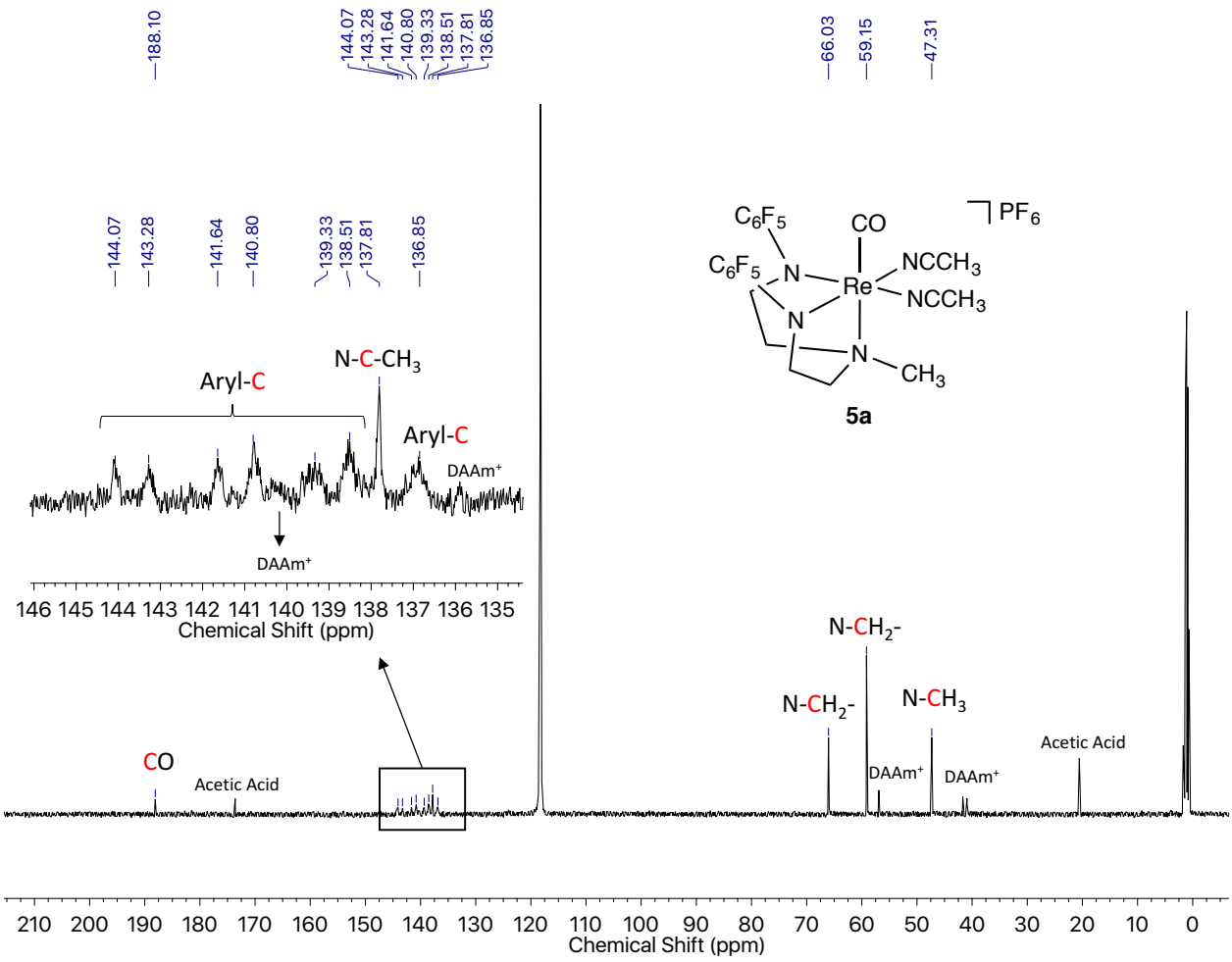


Figure S32. ^{13}C NMR (101 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][PF_6]$ (**5a**). Residual acetic acid and $[DAAm\text{-}C_6F_5]^+$ peaks observed at 173.60 ppm and 20.56 ppm, and 140.16 ppm, 135.94 ppm, 56.90 ppm, 41.68 ppm and 40.93 ppm respectively.

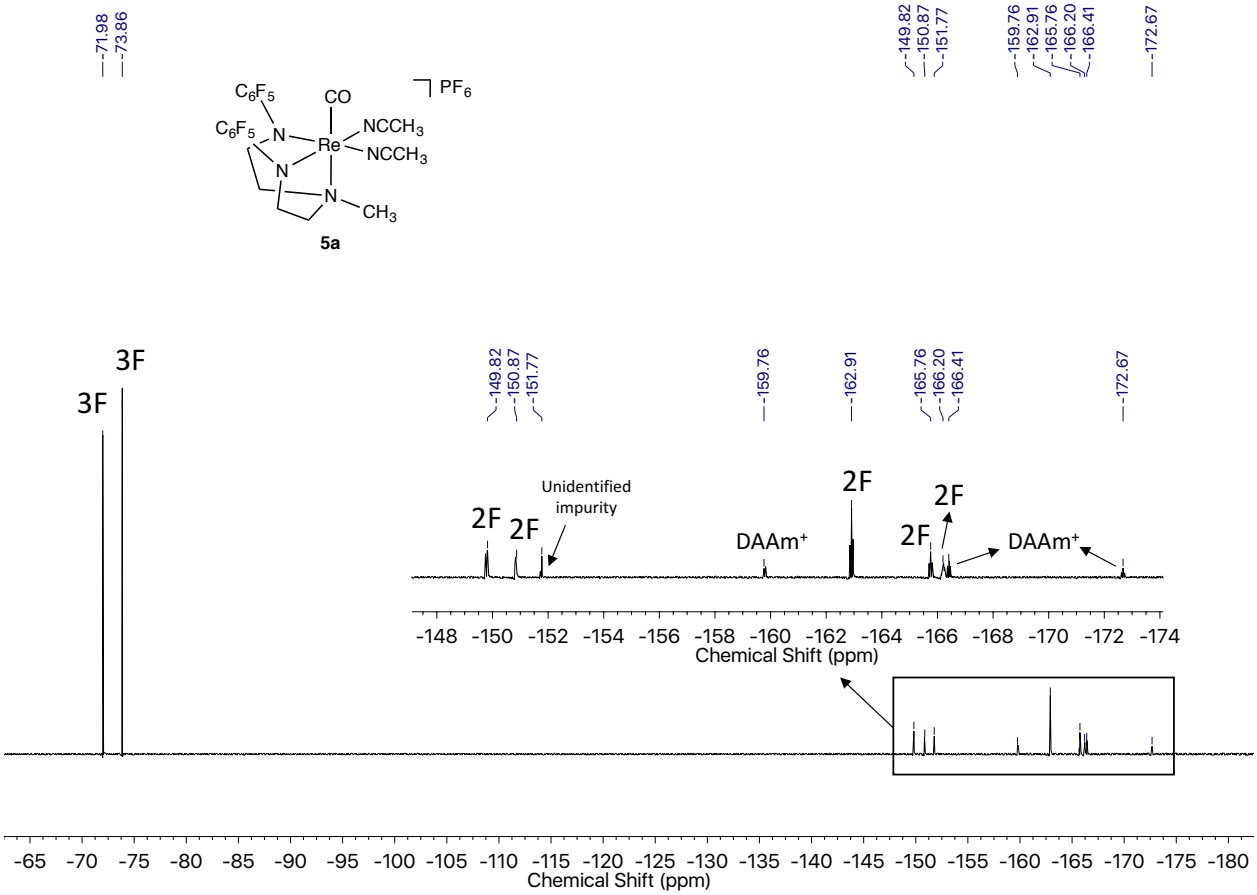


Figure S33. ^{19}F NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][PF_6]$ (**5a**). Residual unidentified impurity and $[DAAm\text{-}C_6F_5]^+$ peaks observed at -151.77 ppm, -159.83 ppm, -166.49 ppm and -172.85 ppm respectively.

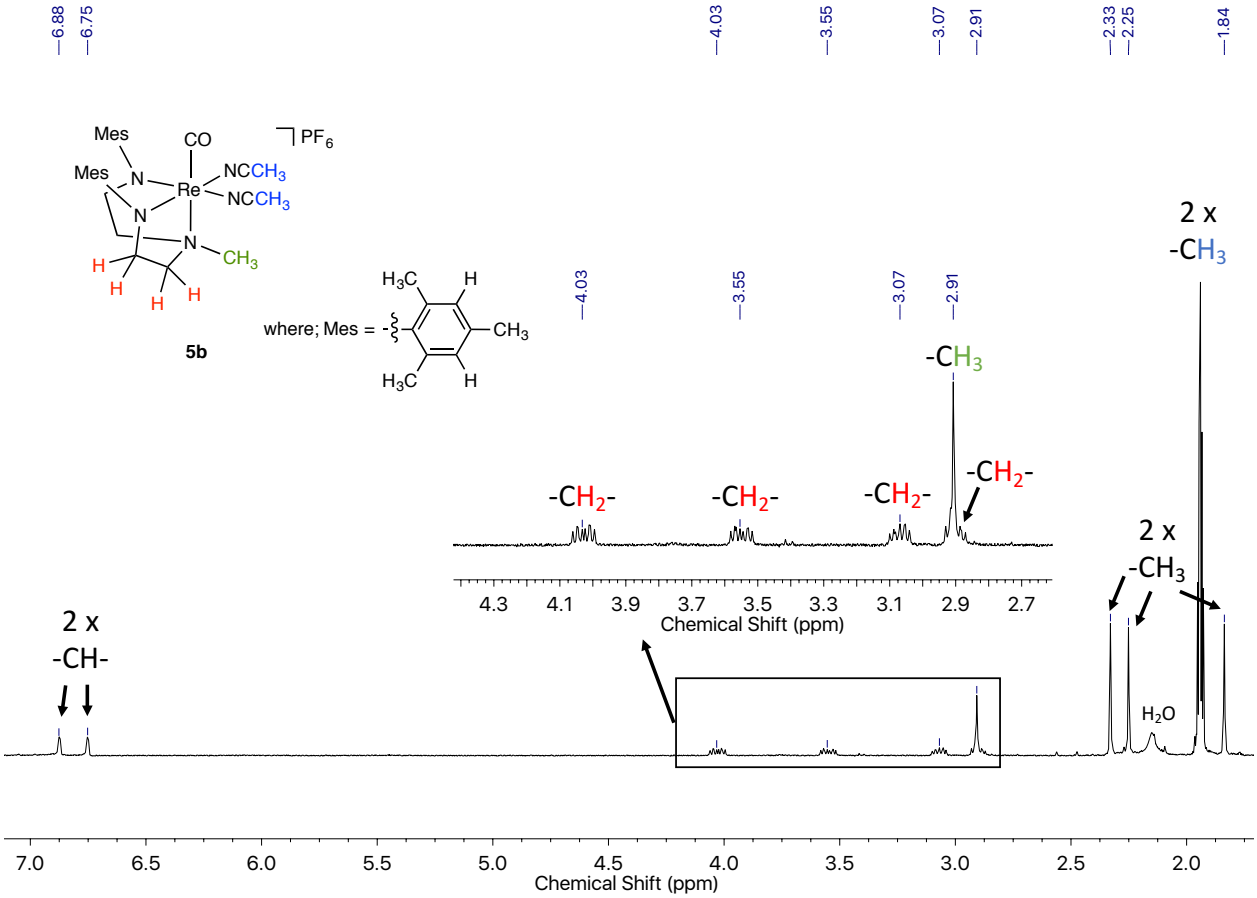


Figure S34. ^{1}H NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][PF_6]$ (**5b**).

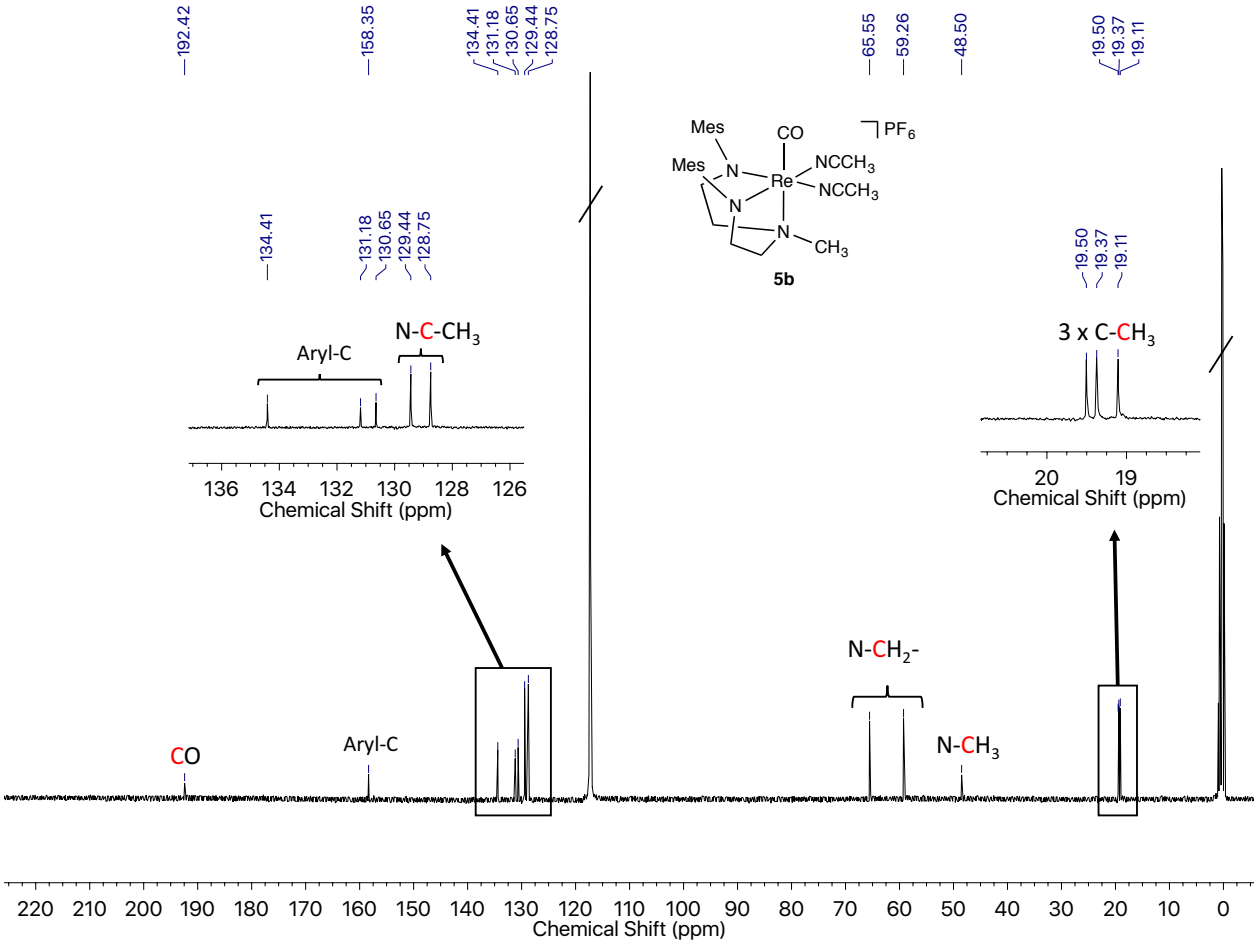


Figure S35. ^{13}C NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][PF_6]$ (**5b**).

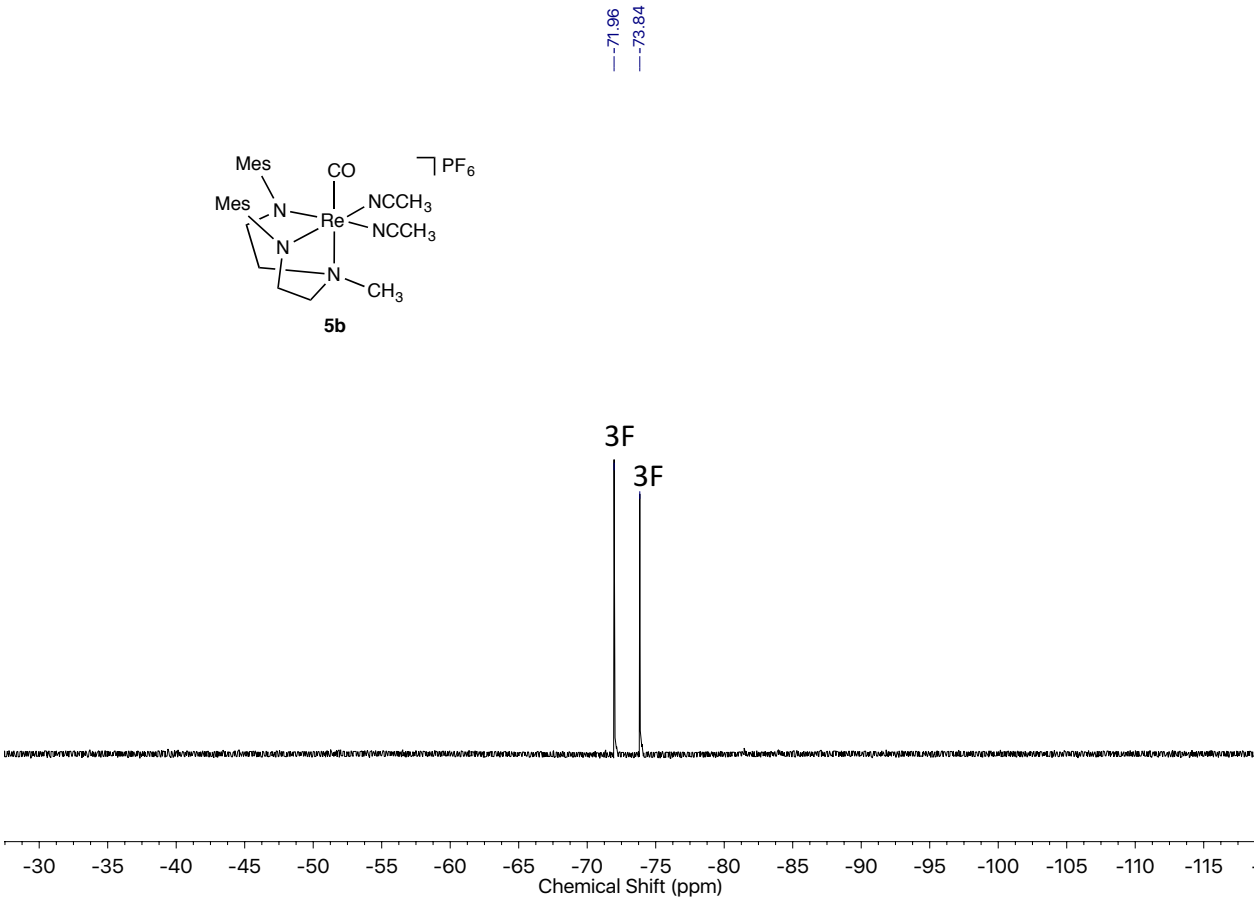


Figure S36. ^{19}F NMR (376 MHz, CD_3CN) spectrum of $[(DAAm\text{-}Mes)Re(CO)(NCCH_3)_2][PF_6]$ (**5b**).

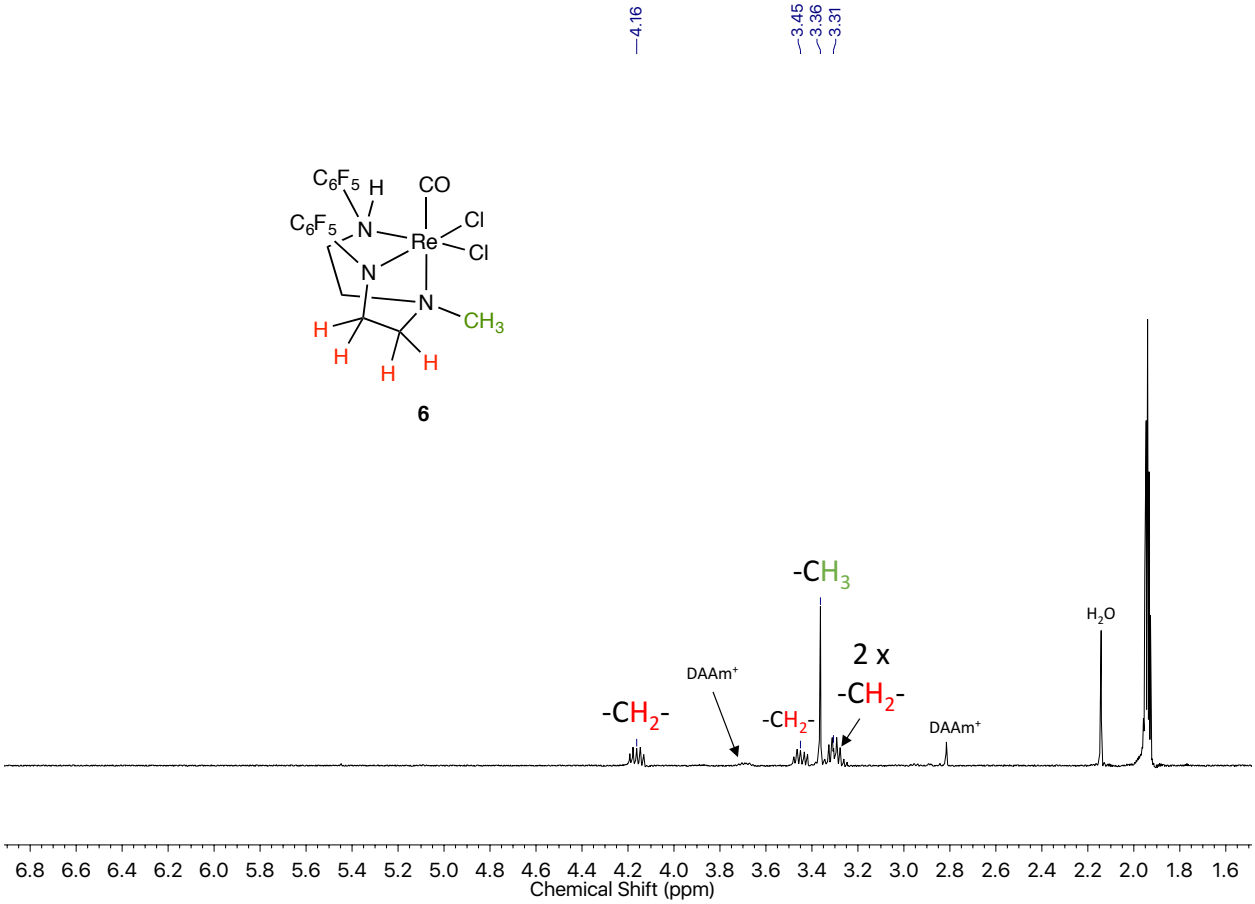


Figure S37. ^{1}H NMR (400 MHz, CD_3CN) spectrum of [(DAmA-C_6F_5)Re(CO)$(Cl)_2$] (**6**). Residual [DAAm-C_6F_5]$^{+}$ and H_2O peaks observed at 3.69 ppm, 2.82 ppm and 2.14 ppm respectively.

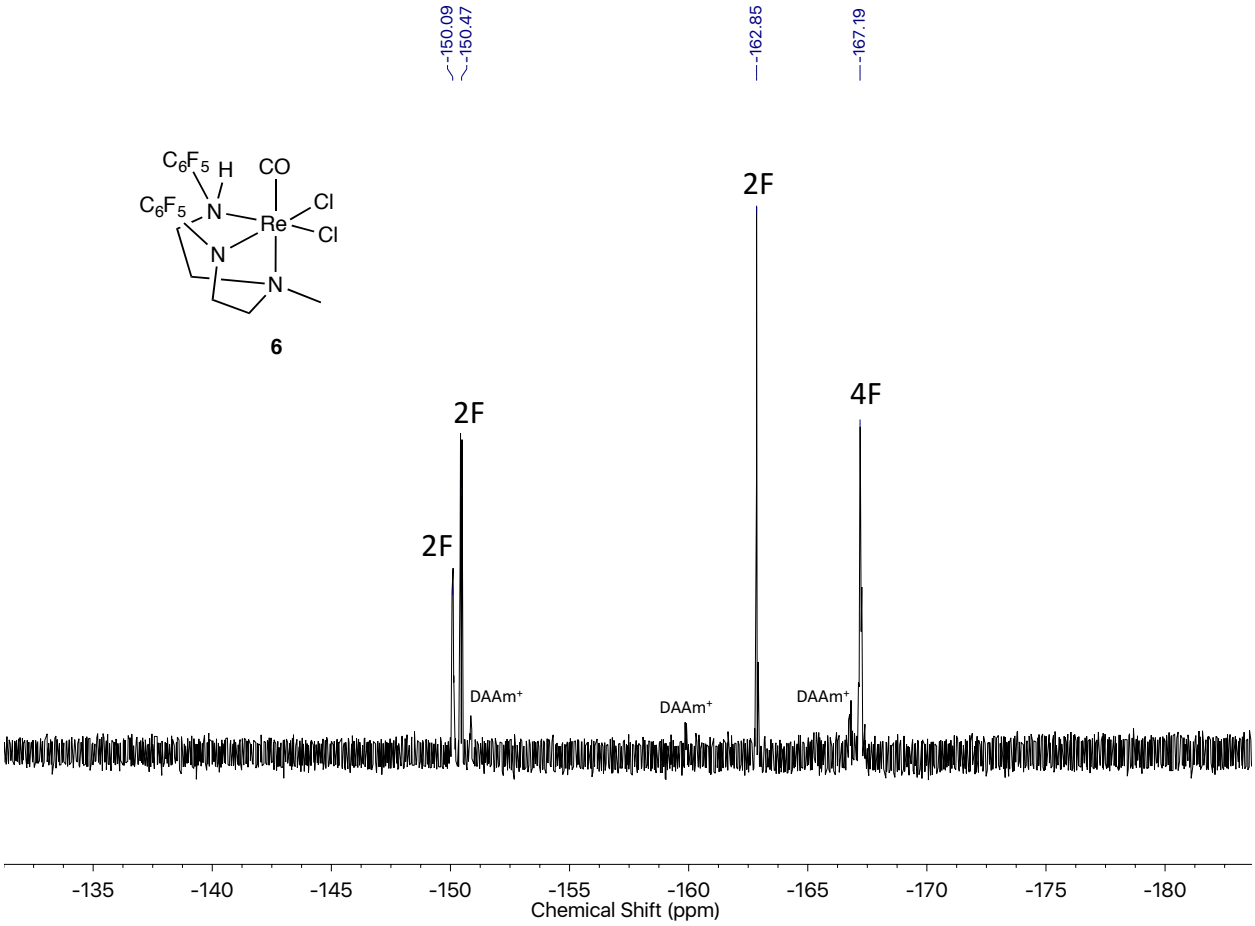


Figure S38. ^{19}F NMR (376 MHz, CD_3CN) spectrum of $[(DAmA\text{-}C_6F_5)Re(CO)(Cl)_2]$ (**6**). Residual $[DAAm\text{-}C_6F_5]^+$ peaks observed at -150.87 ppm, -159.92 ppm and -166.81 ppm.