Electronic Supplementary Material (ESI) for Dalton Transactions.

Influence of Bidentate Ligand Donor Types on the Formation and Stability in 2+1 *fac*-$[M^{I}(CO)_3]^+$ (M = Re, ^{99m}Tc) Complexes

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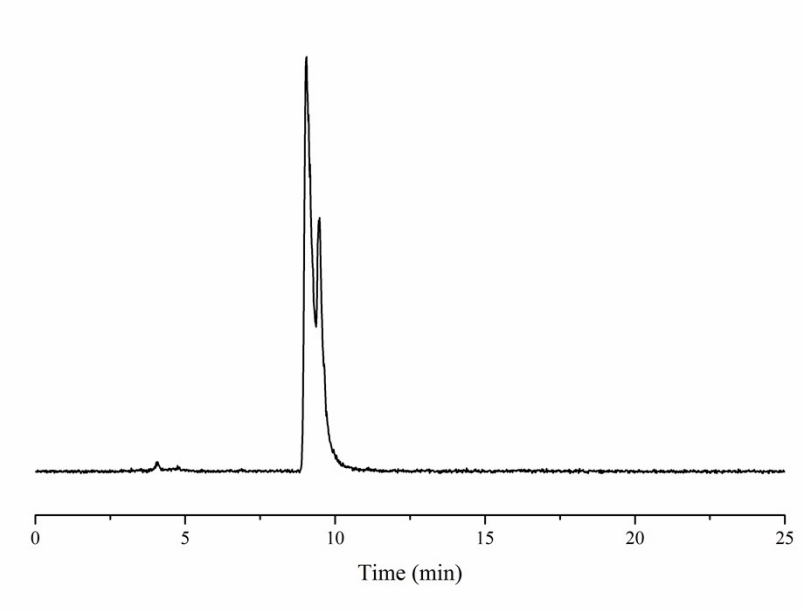

Figure 1. Reaction of *fac*-[^{99m}Tc^{I}(CO)$_3$(**L$_1$**)(OH$_2$)]$^+$ (**1'**) (*left*) with pyridine to form *fac*-[^{99m}Tc^{I}(CO)$_3$(**L$_1$**)(py)]$^+$ (**1a'**) (*right*).

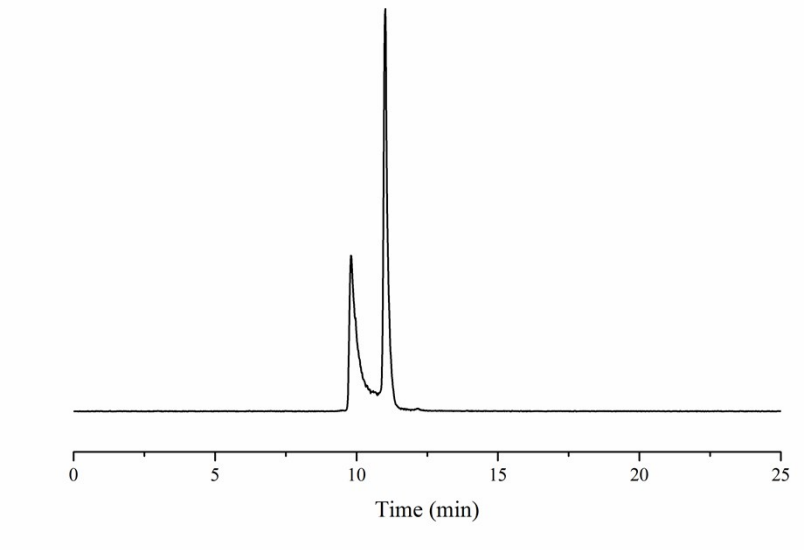

Figure 2. Radio-HPLC chromatogram of the reaction of *fac*-[^{99m}Tc^{I}(CO)$_3$(**L$_1$**)(OH$_2$)]$^+$ (**1'**) with cysteine (1 mM) at 37 °C for 1 h. Retention time for *fac*-[^{99m}Tc^{I}(CO)$_3$(cysteine)] is 4.45 min.

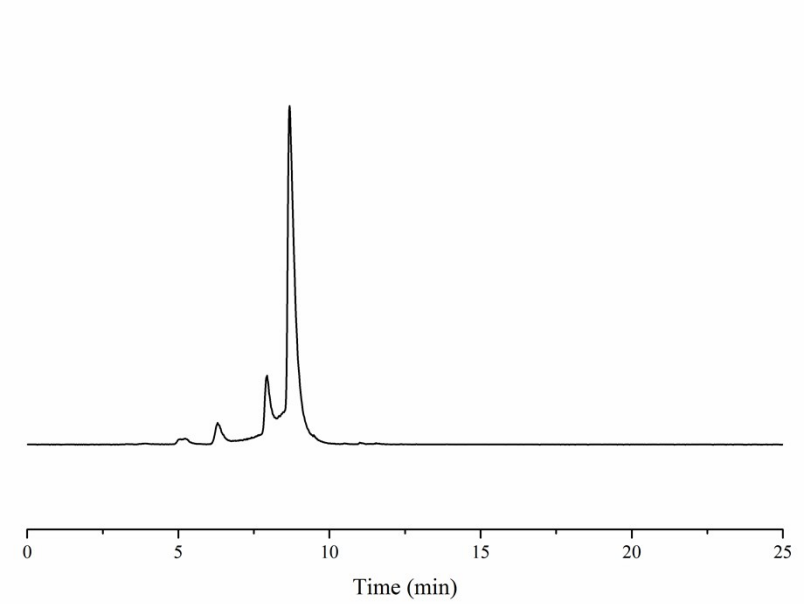

Figure 3. Radio-HPLC chromatogram of the reaction of bipy complex **1'** with histidine (1 mM) at 37 °C for 1 h. Retention time for *fac*-[$^{99m}Tc^{I}(CO)_3$(histidine)] is 5.00 min.

Table 1. Retention times of Re and ^{99m}Tc 2 + 1 complexes.

	Bidentate (L)	Complex	Re t_R (min)	Tc t_R (min)
		$[Tc(OH_2)_3(CO)_3]^+$		4.10
		$[Tc(CO)_3(cys)]$		4.45
		$[Tc(CO)_3(his)]$		5.00
Neutral Ligands	**L_1**	**1a**	8.11	8.4
		1b	9.50	9.72
		1c	10.86	11.08
		1d	8.96	9.15
	L_2	**2a**	8.61	8.72
		2b	9.87	10.1
		2c	11.57	11.83
		2d	9.17	9.28
	L_3	**3a**	13.12	13.26
		3b	13.71	13.75
		3c	14.31	11.41
		3d	13.01	13.25
Anionic Ligands	**L_4**	**4a**	11.47	11.88
		4b	13.07	13.25
		4c	14.03	14.55
		4d	10.51	10.95
	L_5	**5a**	12.02	12.52
		5b	13.52	13.60
		5c	14.32	14.55
		5d	11.46	11.90
	L_6	**6a**	16.36	17.07
		6b	17.12	17.87
		6c	17.32	18.2
		6d	15.31	16.12

Table 2. (U)B3LYP/aug-cc-PVDZ optimized structure of DMAP

N	-1.548054	1.005085	-0.170635
N	-1.970873	5.195842	0.335657
C	-0.898566	1.892291	-0.939080
H	-0.243356	1.469035	-1.709168
C	-1.001033	3.276614	-0.829462
H	-0.431002	3.904704	-1.512599
C	-1.841738	3.834629	0.164205
C	-2.539073	2.903780	0.972155
H	-3.221671	3.228222	1.756314
C	-2.350463	1.540339	0.761632
H	-2.892080	0.826962	1.393475
C	-1.341601	6.104944	-0.605936
H	-0.249313	5.953460	-0.641593
H	-1.523249	7.138525	-0.284663
H	-1.731970	5.990419	-1.635879
C	-2.948104	5.715515	1.275874
H	-3.987058	5.443766	1.005666
H	-2.876815	6.810409	1.300862
H	-2.758648	5.345178	2.297769

Table 3. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **1c**.

Re	8.204861	9.282581	10.654620
O	10.097820	10.710211	12.639968
N	7.030084	8.413244	9.003336
C	6.121032	7.426981	9.155305
H	5.965679	7.071072	10.173803
O	7.253287	7.356873	12.879296
N	8.749595	10.440334	8.858663
C	5.403794	6.887506	8.092190
H	4.678130	6.095186	8.277914
O	5.940656	11.256611	11.402492
N	9.835493	7.855123	10.034685
C	5.634988	7.384724	6.808819
H	5.094879	6.987282	5.948056
N	12.954315	5.146937	9.157666
C	6.563018	8.410308	6.643927
H	6.746003	8.818798	5.651596
C	7.246728	8.916803	7.756238
C	8.202558	10.043611	7.675818
C	8.509502	10.704982	6.480157
H	8.068298	10.377634	5.540343
C	9.376293	11.795274	6.494041
H	9.617398	12.318793	5.567550
C	9.917229	12.208299	7.712448
H	10.589525	13.064005	7.780553
C	9.579939	11.504608	8.864287
H	9.975831	11.798546	9.836405
C	11.110725	8.264459	9.818800
H	11.294134	9.336228	9.901967
C	12.163322	7.421627	9.522943
H	13.148233	7.860285	9.378816
C	11.951184	6.017363	9.430941
C	10.611398	5.592098	9.653535
H	10.332840	4.541280	9.615727
C	9.628808	6.517459	9.943501
H	8.610720	6.172809	10.127773
C	14.318054	5.633232	8.964628
H	14.379908	6.333875	8.114969
H	14.974267	4.782355	8.750658
H	14.699173	6.142112	9.866345
C	12.688184	3.711813	9.101779
H	12.314018	3.330302	10.067053
H	13.618811	3.184437	8.864718
H	11.949545	3.468772	8.319481
C	9.377526	10.161767	11.918470

C	7.622885	8.093270	12.066098
C	6.786912	10.519163	11.127933

Table 4. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **2c**.

Re	9.331114	2.945660	5.594564
O	9.205734	1.316905	2.970575
O	6.826445	4.564763	4.785284
O	11.133270	5.036615	4.198695
N	9.652590	3.846230	7.559488
N	10.746506	4.036717	9.478474
N	11.082350	1.895176	6.494667
N	8.067873	1.424951	6.679894
N	5.477349	-1.393153	8.462761
C	9.097289	4.879860	8.266864
H	8.273993	5.470619	7.877314
C	9.767165	5.005773	9.460001
H	9.636027	5.698417	10.285199
C	10.652762	3.343156	8.302102
C	11.463660	2.255882	7.756817
C	12.542211	1.634003	8.397557
H	12.825490	1.927546	9.404906
C	13.275966	0.630236	7.753660
C	12.877050	0.287728	6.453172
H	13.406941	-0.478441	5.884972
C	11.792892	0.932893	5.872687
H	11.474162	0.683626	4.860260
C	7.090019	1.788506	7.546046
H	6.997051	2.856471	7.745457
C	6.222473	0.906698	8.160366
H	5.466163	1.311368	8.829397
C	6.317181	-0.488515	7.898649
C	7.351398	-0.866826	6.997904
H	7.514231	-1.905880	6.720131
C	8.166825	0.094979	6.434699
H	8.942129	-0.208979	5.730538
C	4.410158	-0.948857	9.354704
H	4.813811	-0.426476	10.238504
H	3.849270	-1.822606	9.704775
H	3.705796	-0.272368	8.841034
C	5.597579	-2.811205	8.135981
H	5.439686	-2.995576	7.059547
H	4.838708	-3.372349	8.692585
H	6.588194	-3.207438	8.416182
C	9.232527	1.945502	3.944146
C	7.766295	3.949777	5.064981
C	10.455367	4.255178	4.715868
C	11.690487	3.859616	10.580499
H	11.589002	2.858980	11.023552

H	12.722559	4.015057	10.236230
H	11.462276	4.607262	11.349002
C	14.447422	-0.042002	8.413953
H	15.378011	0.182658	7.866913
H	14.576066	0.283671	9.455271
H	14.326663	-1.136923	8.404335

Table 5. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **3c**

Re	-1.45845	-1.20178	-0.57158
O	-4.50659	-1.53178	-0.18661
N	0.70347	-0.92873	-0.47893
C	1.29125	-1.03387	0.75475
O	-1.82213	-0.7652	-3.61076
N	1.60593	-0.70435	-1.39949
C	2.65215	-0.86812	0.56301
H	3.50197	-0.86901	1.23662
O	-1.19404	-4.24693	-1.05567
N	2.78543	-0.6688	-0.77223
C	0.42993	-1.3013	1.9004
N	-0.89224	-1.42765	1.5873
C	0.89981	-1.44512	3.20902
H	1.9645	-1.33665	3.41899
N	-1.57066	1.01206	-0.18555
C	-0.0039	-1.73396	4.22928
H	0.34265	-1.85369	5.25706
N	-2.0217	5.18213	0.28834
C	-1.35614	-1.87508	3.90915
H	-2.10076	-2.11109	4.66981
C	-1.75462	-1.71505	2.58477
H	-2.8	-1.82889	2.2968
C	4.02617	-0.51544	-1.57313
H	3.72737	0.06495	-2.45567
H	4.32186	-1.51971	-1.91027
C	5.13641	0.14873	-0.79691
C	6.18495	-0.61748	-0.2675
H	6.20404	-1.69882	-0.4284
C	7.21704	-0.00408	0.44964
H	8.0336	-0.60821	0.84988
C	7.20709	1.37972	0.64222
H	8.01602	1.86029	1.19617
C	6.16602	2.15133	0.11339
H	6.1637	3.23448	0.25121
C	5.13664	1.53967	-0.6038
H	4.33318	2.14844	-1.0274
C	-0.90511	1.90264	-0.96304
H	-0.25738	1.47907	-1.73197
C	-1.01293	3.27428	-0.84265
H	-0.44286	3.8967	-1.52896
C	-1.8671	3.84151	0.14381
C	-2.55291	2.90305	0.96416
H	-3.23503	3.22245	1.74898
C	-2.37787	1.5482	0.76256

H	-2.92461	0.84045	1.38681
C	-1.32161	6.10297	-0.60237
H	-0.22717	5.99066	-0.5206
H	-1.57791	7.132	-0.32669
H	-1.61041	5.94824	-1.65603
C	-2.93256	5.71233	1.29901
H	-3.97096	5.38267	1.12388
H	-2.9117	6.80716	1.26054
H	-2.63418	5.40113	2.31435
C	-3.3671	-1.41121	-0.35782
C	-1.70228	-0.93318	-2.47392
C	-1.297	-3.10903	-0.87875

Table 6. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **4c**.

Re	3.913803	8.909191	2.655596
O	3.876834	9.979374	4.505198
O	2.822117	10.254158	6.482593
O	3.715070	6.998561	0.248582
O	2.349203	11.043856	1.064067
O	6.575671	10.073804	1.597997
N	2.079538	8.201926	3.684336
N	5.018994	7.390744	3.916731
N	7.233107	4.629392	6.217089
C	2.927722	9.759648	5.368969
C	1.876483	8.764959	4.896580
C	0.764470	8.458634	5.679953
H	0.676100	8.955882	6.645992
C	-0.173011	7.546516	5.198945
H	-1.056421	7.290612	5.787840
C	0.037510	6.967056	3.943689
H	-0.668351	6.250818	3.520865
C	1.173140	7.318819	3.218826
H	1.373559	6.897219	2.232689
C	3.818917	7.748864	1.139111
C	2.938999	10.245189	1.664330
C	5.574673	9.634497	1.985384
C	5.748759	7.776906	4.990838
H	5.713360	8.841423	5.224617
C	6.491125	6.908996	5.772103
H	7.045066	7.323568	6.611724
C	6.516965	5.522577	5.472885
C	5.750563	5.125900	4.345749
H	5.703620	4.088529	4.021076
C	5.044501	6.070872	3.622667
H	4.470439	5.761796	2.748221
C	8.018489	5.089988	7.353781
H	8.802437	5.805339	7.048355
H	8.508097	4.228999	7.824161
H	7.385710	5.579944	8.114003
C	7.244612	3.219562	5.855266
H	6.230746	2.783015	5.881696
H	7.867759	2.670024	6.570773
H	7.660564	3.057642	4.844925

Table 7. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **5c**.

Re	4.675059	2.587188	4.499148
O	2.934184	2.325209	3.231109
N	3.026396	2.074355	5.872139
C	2.809178	1.849904	7.208640
H	3.601681	1.939983	7.945644
O	0.714663	1.900699	3.154128
N	0.904167	1.518550	6.137244
C	1.488876	1.502686	7.385886
H	0.927129	1.244744	8.278344
O	6.835339	3.106850	6.628202
N	3.874646	4.696823	4.614845
C	1.865912	1.867659	5.243465
O	6.634091	3.368989	2.241011
N	2.499843	8.686961	4.696003
C	1.781471	2.030441	3.748758
O	5.613774	-0.346459	4.291302
C	-0.490385	1.198318	5.834619
H	-0.629271	1.324821	4.754468
H	-1.155171	1.878185	6.387902
H	-0.705178	0.160054	6.128165
C	3.206174	5.243994	3.572092
H	3.037803	4.582417	2.721781
C	2.738067	6.546304	3.552690
H	2.216213	6.889538	2.661843
C	2.942806	7.394820	4.670814
C	3.640355	6.812717	5.761219
H	3.856209	7.373500	6.668267
C	4.073276	5.500492	5.683477
H	4.620899	5.062185	6.518774
C	1.810963	9.244970	3.540945
H	2.450641	9.244318	2.640546
H	1.529296	10.282354	3.757474
H	0.889900	8.683794	3.305885
C	2.759341	9.525846	5.856593
H	2.306036	9.107235	6.772263
H	2.323926	10.517925	5.687573
H	3.841349	9.653768	6.038727
C	6.040456	2.904254	5.796716
C	5.905142	3.072062	3.094454
C	5.260493	0.756595	4.367066

Table 8. (U)B3LYP/aug-cc-pVDZ-PP/aug-cc-pVDZ optimized structure of **6c**.

Re	7.58655	1.53506	1.84688
O	8.3847	2.97079	3.25714
N	6.73623	0.96971	3.79456
C	7.15778	1.75702	4.82717
O	8.42797	3.6673	5.41429
N	5.86548	0.17627	5.55143
C	6.59066	1.24669	5.9739
H	6.65282	1.55918	7.01046
O	6.08257	-0.51694	0.11052
N	5.95228	0.01382	4.2213
C	8.07416	2.90973	4.52338
O	8.9362	2.74555	-0.65409
N	5.90457	3.03908	1.7427
C	5.03376	-0.7441	6.33703
H	5.64294	-1.12073	7.17071
H	4.82228	-1.58302	5.66047
O	9.92699	-0.45906	2.13286
N	2.84925	5.92724	1.37301
C	3.75926	-0.10453	6.84878
C	2.7811	0.34531	5.94805
H	2.94636	0.2393	4.87285
C	1.60305	0.92549	6.42008
H	0.84661	1.27039	5.71158
C	1.38678	1.05965	7.79653
H	0.46302	1.51168	8.16427
C	2.35451	0.61167	8.69795
H	2.1923	0.71216	9.77327
C	3.53699	0.0334	8.22437
H	4.29131	-0.31748	8.93396
C	6.13554	4.3555	1.96156
H	7.15579	4.61127	2.24977
C	5.16625	5.33719	1.85167
H	5.45747	6.36753	2.04484
C	3.83748	4.9917	1.4957
C	3.60283	3.60968	1.27328
H	2.62159	3.23322	0.99156
C	4.63975	2.70231	1.40372
H	4.45799	1.64266	1.21961
C	3.14697	7.33603	1.58971
H	3.52354	7.52004	2.61086
H	2.2294	7.92152	1.45654
H	3.90026	7.71202	0.87481
C	1.50839	5.52564	0.97538
H	1.49939	5.05026	-0.02173

H	0.86473	6.41245	0.93442
H	1.06406	4.81621	1.6956
C	6.67314	0.26101	0.74804
C	8.4292	2.2826	0.2814
C	9.04873	0.29173	2.02601

Table 9. Crystal data and structure refinement for **1c**.

Identification code	**1c**	
Empirical formula	C21.50 H20 F3 N4 O6.50 Re S	
Formula weight	713.67	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 25.988(6) Å	a= 90°.
	b = 12.548(3) Å	b= 95.685(2)°.
	c = 14.946(3) Å	g = 90°.
Volume	4850.0(19) $Å^3$	
Z	8	
Density (calculated)	1.955 Mg/m^3	
Absorption coefficient	5.169 mm^{-1}	
F(000)	2776	
Crystal size	0.500 x 0.350 x 0.250 mm^3	
Theta range for data collection	1.804 to 27.522°.	
Index ranges	-33<=h<=33, -16<=k<=16, -19<=l<=19	
Reflections collected	26618	
Independent reflections	5537 [R(int) = 0.0357]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.36 and 0.21	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5537 / 0 / 341	
Goodness-of-fit on F^2	1.070	
Final R indices [I>2sigma(I)]	R1 = 0.0289, wR2 = 0.0643	
R indices (all data)	R1 = 0.0428, wR2 = 0.0752	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.409 and -2.319 $e.Å^{-3}$	

Table 10. Bond lengths [Å] and angles [°] for **1c**.

Re(1)-C(18)	1.912(5)	C(6)-C(7)	1.392(6)
Re(1)-C(19)	1.919(4)	C(7)-C(8)	1.383(7)
Re(1)-C(20)	1.939(5)	C(7)-H(7)	0.9500
Re(1)-N(2)	2.164(4)	C(8)-C(9)	1.375(8)
Re(1)-N(1)	2.171(4)	C(8)-H(8)	0.9500
Re(1)-N(3)	2.206(3)	C(9)-C(10)	1.379(7)
S(1)-O(6)	1.437(4)	C(9)-H(9)	0.9500
S(1)-O(4)	1.439(4)	C(10)-H(10)	0.9500
S(1)-O(5)	1.450(4)	C(11)-C(12)	1.367(6)
S(1)-C(21)	1.825(7)	C(11)-H(11)	0.9500
F(1)-C(21)	1.315(7)	C(12)-C(13)	1.414(6)
O(1)-C(18)	1.158(6)	C(12)-H(12)	0.9500
N(1)-C(1)	1.342(6)	C(13)-C(14)	1.417(6)
N(1)-C(5)	1.361(5)	C(14)-C(15)	1.371(6)
C(1)-C(2)	1.385(7)	C(14)-H(14)	0.9500
C(1)-H(1)	0.9500	C(15)-H(15)	0.9500
F(2)-C(21)	1.336(7)	C(16)-H(16A)	0.9800
O(2)-C(19)	1.150(5)	C(16)-H(16B)	0.9800
N(2)-C(10)	1.345(6)	C(16)-H(16C)	0.9800
N(2)-C(6)	1.357(6)	C(17)-H(17A)	0.9800
C(2)-C(3)	1.382(7)	C(17)-H(17B)	0.9800
C(2)-H(2)	0.9500	C(17)-H(17C)	0.9800
F(3)-C(21)	1.329(6)	C(1M)-O(1M)	1.020(16)
O(3)-C(20)	1.137(6)	C(1M)-O(1M)#1	1.020(16)
N(3)-C(11)	1.353(5)		
N(3)-C(15)	1.358(5)	C(18)-Re(1)-C(19)	87.3(2)
C(3)-C(4)	1.393(7)	C(18)-Re(1)-C(20)	90.2(2)
C(3)-H(3)	0.9500	C(19)-Re(1)-C(20)	88.55(18)
N(4)-C(13)	1.349(6)	C(18)-Re(1)-N(2)	100.77(18)
N(4)-C(16)	1.460(6)	C(19)-Re(1)-N(2)	171.50(17)
N(4)-C(17)	1.460(6)	C(20)-Re(1)-N(2)	93.91(16)
C(4)-C(5)	1.389(6)	C(18)-Re(1)-N(1)	172.54(17)
C(4)-H(4)	0.9500	C(19)-Re(1)-N(1)	96.70(17)
C(5)-C(6)	1.479(6)	C(20)-Re(1)-N(1)	96.14(18)

N(2)-Re(1)-N(1)	74.95(14)	C(3)-C(4)-H(4)	120.2
C(18)-Re(1)-N(3)	91.60(16)	N(1)-C(5)-C(4)	121.2(4)
C(19)-Re(1)-N(3)	93.35(15)	N(1)-C(5)-C(6)	114.9(4)
C(20)-Re(1)-N(3)	177.44(16)	C(4)-C(5)-C(6)	123.8(4)
N(2)-Re(1)-N(3)	83.96(13)	N(2)-C(6)-C(7)	121.6(4)
N(1)-Re(1)-N(3)	81.93(13)	N(2)-C(6)-C(5)	115.6(4)
O(6)-S(1)-O(4)	115.1(3)	C(7)-C(6)-C(5)	122.8(4)
O(6)-S(1)-O(5)	115.0(3)	C(8)-C(7)-C(6)	119.1(5)
O(4)-S(1)-O(5)	114.3(2)	C(8)-C(7)-H(7)	120.5
O(6)-S(1)-C(21)	104.6(3)	C(6)-C(7)-H(7)	120.5
O(4)-S(1)-C(21)	102.4(2)	C(9)-C(8)-C(7)	119.4(5)
O(5)-S(1)-C(21)	103.2(3)	C(9)-C(8)-H(8)	120.3
C(1)-N(1)-C(5)	118.7(4)	C(7)-C(8)-H(8)	120.3
C(1)-N(1)-Re(1)	124.4(3)	C(8)-C(9)-C(10)	118.9(5)
C(5)-N(1)-Re(1)	116.5(3)	C(8)-C(9)-H(9)	120.6
N(1)-C(1)-C(2)	122.6(4)	C(10)-C(9)-H(9)	120.6
N(1)-C(1)-H(1)	118.7	N(2)-C(10)-C(9)	122.9(5)
C(2)-C(1)-H(1)	118.7	N(2)-C(10)-H(10)	118.5
C(10)-N(2)-C(6)	118.1(4)	C(9)-C(10)-H(10)	118.5
C(10)-N(2)-Re(1)	125.3(3)	N(3)-C(11)-C(12)	124.5(4)
C(6)-N(2)-Re(1)	116.6(3)	N(3)-C(11)-H(11)	117.8
C(3)-C(2)-C(1)	119.2(4)	C(12)-C(11)-H(11)	117.8
C(3)-C(2)-H(2)	120.4	C(11)-C(12)-C(13)	120.6(4)
C(1)-C(2)-H(2)	120.4	C(11)-C(12)-H(12)	119.7
C(11)-N(3)-C(15)	115.2(4)	C(13)-C(12)-H(12)	119.7
C(11)-N(3)-Re(1)	122.9(3)	N(4)-C(13)-C(12)	123.5(4)
C(15)-N(3)-Re(1)	121.9(3)	N(4)-C(13)-C(14)	121.4(4)
C(2)-C(3)-C(4)	118.6(4)	C(12)-C(13)-C(14)	115.1(4)
C(2)-C(3)-H(3)	120.7	C(15)-C(14)-C(13)	120.1(4)
C(4)-C(3)-H(3)	120.7	C(15)-C(14)-H(14)	120.0
C(13)-N(4)-C(16)	122.2(4)	C(13)-C(14)-H(14)	120.0
C(13)-N(4)-C(17)	121.7(4)	N(3)-C(15)-C(14)	124.5(4)
C(16)-N(4)-C(17)	115.1(4)	N(3)-C(15)-H(15)	117.7
C(5)-C(4)-C(3)	119.6(4)	C(14)-C(15)-H(15)	117.7
C(5)-C(4)-H(4)	120.2	N(4)-C(16)-H(16A)	109.5

N(4)-C(16)-H(16B)	109.5	O(1)-C(18)-Re(1)	178.2(4)
H(16A)-C(16)-H(16B)	109.5	O(2)-C(19)-Re(1)	178.4(5)
N(4)-C(16)-H(16C)	109.5	O(3)-C(20)-Re(1)	179.0(4)
H(16A)-C(16)-H(16C)	109.5	F(1)-C(21)-F(3)	108.1(5)
H(16B)-C(16)-H(16C)	109.5	F(1)-C(21)-F(2)	107.4(6)
N(4)-C(17)-H(17A)	109.5	F(3)-C(21)-F(2)	108.3(4)
N(4)-C(17)-H(17B)	109.5	F(1)-C(21)-S(1)	111.0(4)
H(17A)-C(17)-H(17B)	109.5	F(3)-C(21)-S(1)	110.9(5)
N(4)-C(17)-H(17C)	109.5	F(2)-C(21)-S(1)	111.0(4)
H(17A)-C(17)-H(17C)	109.5	O(1M)-C(1M)-O(1M)#1	150(3)
H(17B)-C(17)-H(17C)	109.5		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2

Table 11. Crystal data and structure refinement for **2c**.

Identification code	**2c**	
Empirical formula	C21 H23 F6 N5 O4 P Re	
Formula weight	740.61	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 28.753(6) Å	a= 90°.
	b = 14.023(3) Å	b= 113.768(2)°.
	c = 14.055(3) Å	g = 90°.
Volume	5186.0(19) $Å^3$	
Z	8	
Density (calculated)	1.897 Mg/m^3	
Absorption coefficient	4.829 mm^{-1}	
F(000)	2880	
Crystal size	0.500 x 0.050 x 0.020 mm^3	
Theta range for data collection	1.548 to 27.471°.	
Index ranges	-37<=h<=37, -17<=k<=18, -18<=l<=18	
Reflections collected	29174	
Independent reflections	5878 [R(int) = 0.0409]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.91 and 0.62	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5878 / 465 / 461	
Goodness-of-fit on F^2	1.145	
Final R indices [I>2sigma(I)]	R1 = 0.0414, wR2 = 0.0814	
R indices (all data)	R1 = 0.0514, wR2 = 0.0850	
Extinction coefficient	n/a	
Largest diff. peak and hole	2.915 and -3.174 $e.Å^{-3}$	

Table 12. Bond lengths [Å] and angles [°] for **2c**.

Re(1)-C(18)	1.889(7)	C(8)-H(8)	0.9500
Re(1)-C(19)	1.908(6)	C(9)-H(9A)	0.9800
Re(1)-C(17)	1.918(8)	C(9)-H(9B)	0.9800
Re(1)-N(1')	1.98(3)	C(9)-H(9C)	0.9800
Re(1)-N(1)	2.119(10)	N(1')-C(3')	1.26(4)
Re(1)-N(2)	2.210(9)	N(1')-C(1')	1.37(4)
Re(1)-N(4)	2.223(5)	C(1')-C(2')	1.34(5)
Re(1)-N(2')	2.26(3)	C(1')-H(1')	0.9500
P(1)-F(3)	1.583(4)	N(2')-C(8')	1.37(5)
P(1)-F(5)	1.584(5)	N(2')-C(4')	1.41(4)
P(1)-F(4)	1.585(4)	C(2')-N(3')	1.40(4)
P(1)-F(6)	1.589(5)	C(2')-H(2')	0.9500
P(1)-F(2)	1.602(4)	N(3')-C(3')	1.39(4)
P(1)-F(1)	1.606(4)	N(3')-C(9')	1.46(3)
O(1)-C(17)	1.150(9)	C(3')-C(4')	1.46(4)
N(1)-C(3)	1.329(12)	C(4')-C(5')	1.35(3)
N(1)-C(1)	1.366(14)	C(5')-C(6')	1.37(3)
C(1)-C(2)	1.509(19)	C(5')-H(5')	0.9500
C(1)-H(1)	0.9500	C(6')-C(7')	1.65(5)
N(2)-C(4)	1.363(11)	C(6')-H(6')	0.9500
N(2)-C(8)	1.363(13)	C(7')-C(8')	0.96(6)
C(2)-N(3)	1.323(17)	C(7')-H(7')	0.9500
C(2)-H(2)	0.9500	C(8')-H(8')	0.9500
N(3)-C(3)	1.378(11)	C(9')-H(9'1)	0.9800
N(3)-C(9)	1.457(11)	C(9')-H(9'2)	0.9800
C(3)-C(4)	1.461(12)	C(9')-H(9'3)	0.9800
C(4)-C(5)	1.381(11)	O(2S)-O(2S)#1	1.286(15)
C(5)-C(6)	1.373(12)	O(2S)-C(3S)#1	1.286(15)
C(5)-H(5)	0.9500	O(2)-C(18)	1.148(9)
C(6)-C(7)	1.384(15)	O(3)-C(19)	1.152(7)
C(6)-H(6)	0.9500	N(4)-C(14)	1.355(7)
C(7)-C(8)	1.371(15)	N(4)-C(10)	1.355(7)
C(7)-H(7)	0.9500	N(5)-C(12)	1.343(7)

N(5)-C(15)	1.457(8)
N(5)-C(16)	1.466(7)
C(10)-C(11)	1.369(8)
C(10)-H(10)	0.9500
C(11)-C(12)	1.412(8)
C(11)-H(11)	0.9500
C(12)-C(13)	1.416(8)
C(13)-C(14)	1.371(8)
C(13)-H(13)	0.9500
C(14)-H(14)	0.9500
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800

C(18)-Re(1)-C(19)	86.8(3)
C(18)-Re(1)-C(17)	90.6(4)
C(19)-Re(1)-C(17)	88.0(3)
C(18)-Re(1)-N(1')	177.4(8)
C(19)-Re(1)-N(1')	94.4(9)
C(17)-Re(1)-N(1')	87.1(8)
C(18)-Re(1)-N(1)	97.4(4)
C(19)-Re(1)-N(1)	93.8(4)
C(17)-Re(1)-N(1)	171.8(4)
C(18)-Re(1)-N(2)	171.6(4)
C(19)-Re(1)-N(2)	93.4(3)
C(17)-Re(1)-N(2)	97.8(4)
N(1)-Re(1)-N(2)	74.1(3)
C(18)-Re(1)-N(4)	93.2(2)
C(19)-Re(1)-N(4)	179.8(3)
C(17)-Re(1)-N(4)	92.1(3)
N(1')-Re(1)-N(4)	85.6(9)
N(1)-Re(1)-N(4)	86.1(3)
N(2)-Re(1)-N(4)	86.6(3)
C(18)-Re(1)-N(2')	106.3(9)
C(19)-Re(1)-N(2')	92.5(10)
C(17)-Re(1)-N(2')	163.1(9)
N(1')-Re(1)-N(2')	76.0(11)
N(4)-Re(1)-N(2')	87.4(10)
F(3)-P(1)-F(5)	90.8(3)
F(3)-P(1)-F(4)	91.6(2)
F(5)-P(1)-F(4)	90.0(3)
F(3)-P(1)-F(6)	90.6(3)
F(5)-P(1)-F(6)	177.9(3)
F(4)-P(1)-F(6)	91.5(3)
F(3)-P(1)-F(2)	89.2(2)
F(5)-P(1)-F(2)	89.7(3)
F(4)-P(1)-F(2)	179.1(3)
F(6)-P(1)-F(2)	88.8(3)
F(3)-P(1)-F(1)	179.0(3)
F(5)-P(1)-F(1)	89.7(3)
F(4)-P(1)-F(1)	89.3(2)
F(6)-P(1)-F(1)	88.8(3)
F(2)-P(1)-F(1)	89.9(2)
C(3)-N(1)-C(1)	108.1(9)
C(3)-N(1)-Re(1)	117.0(7)
C(1)-N(1)-Re(1)	134.5(7)
N(1)-C(1)-C(2)	107.7(9)
N(1)-C(1)-H(1)	126.1
C(2)-C(1)-H(1)	126.1
C(4)-N(2)-C(8)	116.5(9)
C(4)-N(2)-Re(1)	117.9(6)
C(8)-N(2)-Re(1)	125.6(7)
N(3)-C(2)-C(1)	102.3(11)
N(3)-C(2)-H(2)	128.8
C(1)-C(2)-H(2)	128.8
C(2)-N(3)-C(3)	112.5(9)
C(2)-N(3)-C(9)	118.5(9)

C(3)-N(3)-C(9)	128.9(8)	C(1')-C(2')-H(2')	127.0
N(1)-C(3)-N(3)	109.3(8)	N(3')-C(2')-H(2')	127.0
N(1)-C(3)-C(4)	120.0(8)	C(3')-N(3')-C(2')	104(3)
N(3)-C(3)-C(4)	130.6(9)	C(3')-N(3')-C(9')	131(2)
N(2)-C(4)-C(5)	123.0(9)	C(2')-N(3')-C(9')	125(3)
N(2)-C(4)-C(3)	110.5(7)	N(1')-C(3')-N(3')	113(3)
C(5)-C(4)-C(3)	126.4(10)	N(1')-C(3')-C(4')	124(3)
C(6)-C(5)-C(4)	118.7(9)	N(3')-C(3')-C(4')	124(3)
C(6)-C(5)-H(5)	120.7	C(5')-C(4')-N(2')	117(3)
C(4)-C(5)-H(5)	120.7	C(5')-C(4')-C(3')	135(3)
C(5)-C(6)-C(7)	119.7(10)	N(2')-C(4')-C(3')	109(2)
C(5)-C(6)-H(6)	120.1	C(4')-C(5')-C(6')	121(2)
C(7)-C(6)-H(6)	120.1	C(4')-C(5')-H(5')	119.6
C(8)-C(7)-C(6)	118.8(11)	C(6')-C(5')-H(5')	119.6
C(8)-C(7)-H(7)	120.6	C(5')-C(6')-C(7')	112(3)
C(6)-C(7)-H(7)	120.6	C(5')-C(6')-H(6')	123.8
N(2)-C(8)-C(7)	123.2(10)	C(7')-C(6')-H(6')	123.8
N(2)-C(8)-H(8)	118.4	C(8')-C(7')-C(6')	120(5)
C(7)-C(8)-H(8)	118.4	C(8')-C(7')-H(7')	119.8
N(3)-C(9)-H(9A)	109.5	C(6')-C(7')-H(7')	119.8
N(3)-C(9)-H(9B)	109.5	C(7')-C(8')-N(2')	129(4)
H(9A)-C(9)-H(9B)	109.5	C(7')-C(8')-H(8')	115.4
N(3)-C(9)-H(9C)	109.5	N(2')-C(8')-H(8')	115.4
H(9A)-C(9)-H(9C)	109.5	N(3')-C(9')-H(9'1)	109.5
H(9B)-C(9)-H(9C)	109.5	N(3')-C(9')-H(9'2)	109.5
C(3')-N(1')-C(1')	106(3)	H(9'1)-C(9')-H(9'2)	109.5
C(3')-N(1')-Re(1)	119(2)	N(3')-C(9')-H(9'3)	109.5
C(1')-N(1')-Re(1)	134(2)	H(9'1)-C(9')-H(9'3)	109.5
C(2')-C(1')-N(1')	111(3)	H(9'2)-C(9')-H(9'3)	109.5
C(2')-C(1')-H(1')	124.6	O(2S)#1-O(2S)-C(3S)#1	0.0(6)
N(1')-C(1')-H(1')	124.6	C(14)-N(4)-C(10)	114.6(5)
C(8')-N(2')-C(4')	120(3)	C(14)-N(4)-Re(1)	120.5(4)
C(8')-N(2')-Re(1)	128(2)	C(10)-N(4)-Re(1)	124.7(4)
C(4')-N(2')-Re(1)	113(2)	C(12)-N(5)-C(15)	121.4(5)
C(1')-C(2')-N(3')	106(4)	C(12)-N(5)-C(16)	121.4(5)

C(15)-N(5)-C(16)	116.9(5)	N(5)-C(15)-H(15A)	109.5
N(4)-C(10)-C(11)	124.5(5)	N(5)-C(15)-H(15B)	109.5
N(4)-C(10)-H(10)	117.7	H(15A)-C(15)-H(15B)	109.5
C(11)-C(10)-H(10)	117.7	N(5)-C(15)-H(15C)	109.5
C(10)-C(11)-C(12)	120.9(6)	H(15A)-C(15)-H(15C)	109.5
C(10)-C(11)-H(11)	119.5	H(15B)-C(15)-H(15C)	109.5
C(12)-C(11)-H(11)	119.5	N(5)-C(16)-H(16A)	109.5
N(5)-C(12)-C(11)	123.0(6)	N(5)-C(16)-H(16B)	109.5
N(5)-C(12)-C(13)	122.6(5)	H(16A)-C(16)-H(16B)	109.5
C(11)-C(12)-C(13)	114.4(5)	N(5)-C(16)-H(16C)	109.5
C(14)-C(13)-C(12)	120.4(5)	H(16A)-C(16)-H(16C)	109.5
C(14)-C(13)-H(13)	119.8	H(16B)-C(16)-H(16C)	109.5
C(12)-C(13)-H(13)	119.8	O(1)-C(17)-Re(1)	177.4(10)
N(4)-C(14)-C(13)	124.9(6)	O(2)-C(18)-Re(1)	178.1(7)
N(4)-C(14)-H(14)	117.6	O(3)-C(19)-Re(1)	177.4(6)
C(13)-C(14)-H(14)	117.6		

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+1/2

Table 13. Crystal data and structure refinement for **3c**.

Identification code	**3c**	
Empirical formula	C25 H22 F3 N6 O6 Re S	
Formula weight	777.74	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.3595(8) Å	a= 86.3080(10)°.
	b = 11.0688(10) Å	b= 87.1240(10)°.
	c = 14.8076(13) Å	g = 86.9900(10)°.
Volume	1363.9(2) $Å^3$	
Z	2	
Density (calculated)	1.894 Mg/m^3	
Absorption coefficient	4.604 mm^{-1}	
F(000)	760	
Crystal size	0.550 x 0.350 x 0.150 mm^3	
Theta range for data collection	1.846 to 27.661°.	
Index ranges	-10<=h<=10, -14<=k<=14, -19<=l<=19	
Reflections collected	15308	
Independent reflections	6193 [R(int) = 0.0231]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.55 and 0.35	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6193 / 0 / 381	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0187, wR2 = 0.0438	
R indices (all data)	R1 = 0.0206, wR2 = 0.0446	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.247 and -0.746 $e.Å^{-3}$	

Table 14. Bond lengths [Å] and angles [°] for **3c**.

Re(1)-C(24)	1.915(3)	N(6)-C(20)	1.461(3)
Re(1)-C(23)	1.924(2)	N(6)-C(21)	1.461(3)
Re(1)-C(22)	1.929(2)	C(6)-C(7)	1.383(3)
Re(1)-N(1)	2.1401(19)	C(6)-H(6)	0.9500
Re(1)-N(4)	2.1939(19)	C(7)-H(7)	0.9500
Re(1)-N(5)	2.206(2)	C(8)-C(9)	1.512(3)
S(1)-O(6)	1.4345(19)	C(8)-H(8A)	0.9900
S(1)-O(5)	1.441(2)	C(8)-H(8B)	0.9900
S(1)-O(4)	1.450(2)	C(9)-C(14)	1.386(4)
S(1)-C(25)	1.823(3)	C(9)-C(10)	1.388(4)
F(1)-C(25)	1.332(3)	C(10)-C(11)	1.389(4)
O(1)-C(22)	1.152(3)	C(10)-H(10)	0.9500
N(1)-N(2)	1.319(3)	C(11)-C(12)	1.375(4)
N(1)-C(1)	1.366(3)	C(11)-H(11)	0.9500
C(1)-C(2)	1.372(3)	C(12)-C(13)	1.381(5)
C(1)-C(3)	1.462(3)	C(12)-H(12)	0.9500
F(2)-C(25)	1.328(3)	C(13)-C(14)	1.384(4)
O(2)-C(23)	1.157(3)	C(13)-H(13)	0.9500
N(2)-N(3)	1.342(3)	C(14)-H(14)	0.9500
C(2)-N(3)	1.348(3)	C(15)-C(16)	1.368(3)
C(2)-H(2)	0.9500	C(15)-H(15)	0.9500
F(3)-C(25)	1.330(3)	C(16)-C(17)	1.416(3)
O(3)-C(24)	1.153(3)	C(16)-H(16)	0.9500
N(3)-C(8)	1.483(3)	C(17)-C(18)	1.423(3)
C(3)-N(4)	1.358(3)	C(18)-C(19)	1.370(3)
C(3)-C(4)	1.387(3)	C(18)-H(18)	0.9500
N(4)-C(7)	1.351(3)	C(19)-H(19)	0.9500
C(4)-C(5)	1.384(3)	C(20)-H(20A)	0.9800
C(4)-H(4)	0.9500	C(20)-H(20B)	0.9800
N(5)-C(15)	1.348(3)	C(20)-H(20C)	0.9800
N(5)-C(19)	1.361(3)	C(21)-H(21A)	0.9800
C(5)-C(6)	1.389(3)	C(21)-H(21B)	0.9800
C(5)-H(5)	0.9500	C(21)-H(21C)	0.9800
N(6)-C(17)	1.346(3)		

C(24)-Re(1)-C(23)	92.05(10)	N(4)-C(3)-C(1)	113.8(2)
C(24)-Re(1)-C(22)	87.71(10)	C(4)-C(3)-C(1)	123.6(2)
C(23)-Re(1)-C(22)	89.61(10)	C(7)-N(4)-C(3)	118.1(2)
C(24)-Re(1)-N(1)	93.70(9)	C(7)-N(4)-Re(1)	124.32(15)
C(23)-Re(1)-N(1)	97.47(8)	C(3)-N(4)-Re(1)	117.52(15)
C(22)-Re(1)-N(1)	172.72(8)	C(5)-C(4)-C(3)	118.7(2)
C(24)-Re(1)-N(4)	94.09(8)	C(5)-C(4)-H(4)	120.7
C(23)-Re(1)-N(4)	170.17(8)	C(3)-C(4)-H(4)	120.7
C(22)-Re(1)-N(4)	98.29(8)	C(15)-N(5)-C(19)	115.9(2)
N(1)-Re(1)-N(4)	74.49(7)	C(15)-N(5)-Re(1)	122.12(16)
C(24)-Re(1)-N(5)	176.11(8)	C(19)-N(5)-Re(1)	121.99(16)
C(23)-Re(1)-N(5)	90.98(9)	C(4)-C(5)-C(6)	119.0(2)
C(22)-Re(1)-N(5)	94.74(9)	C(4)-C(5)-H(5)	120.5
N(1)-Re(1)-N(5)	83.49(7)	C(6)-C(5)-H(5)	120.5
N(4)-Re(1)-N(5)	82.59(7)	C(17)-N(6)-C(20)	121.1(2)
O(6)-S(1)-O(5)	115.57(12)	C(17)-N(6)-C(21)	120.9(2)
O(6)-S(1)-O(4)	114.47(13)	C(20)-N(6)-C(21)	117.7(2)
O(5)-S(1)-O(4)	115.23(13)	C(7)-C(6)-C(5)	119.6(2)
O(6)-S(1)-C(25)	103.43(13)	C(7)-C(6)-H(6)	120.2
O(5)-S(1)-C(25)	103.90(13)	C(5)-C(6)-H(6)	120.2
O(4)-S(1)-C(25)	101.66(13)	N(4)-C(7)-C(6)	122.0(2)
N(2)-N(1)-C(1)	110.61(19)	N(4)-C(7)-H(7)	119.0
N(2)-N(1)-Re(1)	131.86(15)	C(6)-C(7)-H(7)	119.0
C(1)-N(1)-Re(1)	117.45(15)	N(3)-C(8)-C(9)	109.67(19)
N(1)-C(1)-C(2)	107.4(2)	N(3)-C(8)-H(8A)	109.7
N(1)-C(1)-C(3)	116.7(2)	C(9)-C(8)-H(8A)	109.7
C(2)-C(1)-C(3)	135.9(2)	N(3)-C(8)-H(8B)	109.7
N(1)-N(2)-N(3)	104.98(18)	C(9)-C(8)-H(8B)	109.7
N(3)-C(2)-C(1)	104.2(2)	H(8A)-C(8)-H(8B)	108.2
N(3)-C(2)-H(2)	127.9	C(14)-C(9)-C(10)	119.3(2)
C(1)-C(2)-H(2)	127.9	C(14)-C(9)-C(8)	120.0(2)
N(2)-N(3)-C(2)	112.81(19)	C(10)-C(9)-C(8)	120.6(2)
N(2)-N(3)-C(8)	118.84(19)	C(9)-C(10)-C(11)	119.6(3)
C(2)-N(3)-C(8)	128.0(2)	C(9)-C(10)-H(10)	120.2
N(4)-C(3)-C(4)	122.7(2)	C(11)-C(10)-H(10)	120.2

C(12)-C(11)-C(10)	121.0(3)	N(5)-C(19)-C(18)	123.7(2)
C(12)-C(11)-H(11)	119.5	N(5)-C(19)-H(19)	118.1
C(10)-C(11)-H(11)	119.5	C(18)-C(19)-H(19)	118.1
C(11)-C(12)-C(13)	119.3(3)	N(6)-C(20)-H(20A)	109.5
C(11)-C(12)-H(12)	120.3	N(6)-C(20)-H(20B)	109.5
C(13)-C(12)-H(12)	120.3	H(20A)-C(20)-H(20B)	109.5
C(12)-C(13)-C(14)	120.3(3)	N(6)-C(20)-H(20C)	109.5
C(12)-C(13)-H(13)	119.8	H(20A)-C(20)-H(20C)	109.5
C(14)-C(13)-H(13)	119.8	H(20B)-C(20)-H(20C)	109.5
C(13)-C(14)-C(9)	120.4(3)	N(6)-C(21)-H(21A)	109.5
C(13)-C(14)-H(14)	119.8	N(6)-C(21)-H(21B)	109.5
C(9)-C(14)-H(14)	119.8	H(21A)-C(21)-H(21B)	109.5
N(5)-C(15)-C(16)	124.5(2)	N(6)-C(21)-H(21C)	109.5
N(5)-C(15)-H(15)	117.8	H(21A)-C(21)-H(21C)	109.5
C(16)-C(15)-H(15)	117.8	H(21B)-C(21)-H(21C)	109.5
C(15)-C(16)-C(17)	120.4(2)	O(1)-C(22)-Re(1)	177.7(2)
C(15)-C(16)-H(16)	119.8	O(2)-C(23)-Re(1)	176.8(2)
C(17)-C(16)-H(16)	119.8	O(3)-C(24)-Re(1)	179.3(2)
N(6)-C(17)-C(16)	123.0(2)	F(2)-C(25)-F(3)	107.2(2)
N(6)-C(17)-C(18)	122.0(2)	F(2)-C(25)-F(1)	107.6(2)
C(16)-C(17)-C(18)	115.0(2)	F(3)-C(25)-F(1)	107.5(2)
C(19)-C(18)-C(17)	120.5(2)	F(2)-C(25)-S(1)	111.1(2)
C(19)-C(18)-H(18)	119.7	F(3)-C(25)-S(1)	111.58(19)
C(17)-C(18)-H(18)	119.7	F(1)-C(25)-S(1)	111.66(18)

Symmetry transformations used to generate equivalent atoms:

Table 15. Crystal data and structure refinement for **5c**.

Identification code	**5c**	
Empirical formula	C15 H15 N4 O5 Re	
Formula weight	517.51	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C c	
Unit cell dimensions	a = 16.127(2) Å	a= 90°.
	b = 15.010(2) Å	b= 109.0350(10)°.
	c = 7.2212(10) Å	g = 90°.
Volume	1652.4(4) $Å^3$	
Z	4	
Density (calculated)	2.080 Mg/m^3	
Absorption coefficient	7.389 mm^{-1}	
F(000)	992	
Crystal size	0.500 x 0.300 x 0.150 mm^3	
Theta range for data collection	1.904 to 27.451°.	
Index ranges	-20<=h<=20, -19<=k<=19, -9<=l<=9	
Reflections collected	7212	
Independent reflections	3612 [R(int) = 0.0211]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.40 and 0.19	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3612 / 2 / 230	
Goodness-of-fit on F^2	0.888	
Final R indices [I>2sigma(I)]	R1 = 0.0163, wR2 = 0.0373	
R indices (all data)	R1 = 0.0167, wR2 = 0.0374	
Absolute structure parameter	0.012(9)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.940 and -1.027 $e.Å^{-3}$	

Table 16. Bond lengths [Å] and angles [°] for **5c**.

Re(1)-C(13)	1.899(6)	C(9)-H(9)	0.9500
Re(1)-C(15)	1.920(5)	C(10)-H(10)	0.9500
Re(1)-C(14)	1.923(6)	C(11)-H(11A)	0.9800
Re(1)-N(1)	2.152(4)	C(11)-H(11B)	0.9800
Re(1)-O(1)	2.161(4)	C(11)-H(11C)	0.9800
Re(1)-N(3)	2.226(4)	C(12)-H(12A)	0.9800
O(1)-C(4)	1.299(6)	C(12)-H(12B)	0.9800
N(1)-C(3)	1.344(6)	C(12)-H(12C)	0.9800
N(1)-C(1)	1.378(6)		
C(1)-C(2)	1.367(7)	C(13)-Re(1)-C(15)	86.8(2)
C(1)-H(1)	0.9500	C(13)-Re(1)-C(14)	88.8(2)
O(2)-C(4)	1.227(6)	C(15)-Re(1)-C(14)	88.7(2)
N(2)-C(3)	1.343(7)	C(13)-Re(1)-N(1)	99.4(2)
N(2)-C(2)	1.372(7)	C(15)-Re(1)-N(1)	95.04(19)
N(2)-C(5)	1.466(7)	C(14)-Re(1)-N(1)	171.16(19)
C(2)-H(2)	0.9500	C(13)-Re(1)-O(1)	173.9(2)
O(3)-C(13)	1.157(7)	C(15)-Re(1)-O(1)	96.59(19)
N(3)-C(6)	1.353(7)	C(14)-Re(1)-O(1)	96.33(19)
N(3)-C(10)	1.374(8)	N(1)-Re(1)-O(1)	75.32(16)
C(3)-C(4)	1.487(7)	C(13)-Re(1)-N(3)	92.3(2)
O(4)-C(14)	1.140(6)	C(15)-Re(1)-N(3)	178.94(19)
N(4)-C(8)	1.357(7)	C(14)-Re(1)-N(3)	90.76(19)
N(4)-C(12)	1.445(7)	N(1)-Re(1)-N(3)	85.58(16)
N(4)-C(11)	1.452(7)	O(1)-Re(1)-N(3)	84.40(15)
O(5)-C(15)	1.148(6)	C(4)-O(1)-Re(1)	119.3(3)
C(5)-H(5A)	0.9800	C(3)-N(1)-C(1)	106.4(4)
C(5)-H(5B)	0.9800	C(3)-N(1)-Re(1)	113.9(3)
C(5)-H(5C)	0.9800	C(1)-N(1)-Re(1)	139.5(3)
C(6)-C(7)	1.366(7)	C(2)-C(1)-N(1)	108.7(4)
C(6)-H(6)	0.9500	C(2)-C(1)-H(1)	125.6
C(7)-C(8)	1.412(7)	N(1)-C(1)-H(1)	125.6
C(7)-H(7)	0.9500	C(3)-N(2)-C(2)	108.0(4)
C(8)-C(9)	1.413(7)	C(3)-N(2)-C(5)	127.7(5)
C(9)-C(10)	1.369(8)	C(2)-N(2)-C(5)	124.3(5)

C(1)-C(2)-N(2)	106.7(5)	C(8)-C(7)-H(7)	119.9
C(1)-C(2)-H(2)	126.7	N(4)-C(8)-C(7)	123.2(5)
N(2)-C(2)-H(2)	126.7	N(4)-C(8)-C(9)	122.3(5)
C(6)-N(3)-C(10)	115.7(5)	C(7)-C(8)-C(9)	114.5(5)
C(6)-N(3)-Re(1)	125.3(3)	C(10)-C(9)-C(8)	122.5(6)
C(10)-N(3)-Re(1)	119.0(4)	C(10)-C(9)-H(9)	118.8
N(2)-C(3)-N(1)	110.2(4)	C(8)-C(9)-H(9)	118.8
N(2)-C(3)-C(4)	130.4(5)	C(9)-C(10)-N(3)	122.0(7)
N(1)-C(3)-C(4)	119.3(4)	C(9)-C(10)-H(10)	119.0
C(8)-N(4)-C(12)	120.7(5)	N(3)-C(10)-H(10)	119.0
C(8)-N(4)-C(11)	120.9(5)	N(4)-C(11)-H(11A)	109.5
C(12)-N(4)-C(11)	118.2(5)	N(4)-C(11)-H(11B)	109.5
O(2)-C(4)-O(1)	126.1(5)	H(11A)-C(11)-H(11B)	109.5
O(2)-C(4)-C(3)	121.7(5)	N(4)-C(11)-H(11C)	109.5
O(1)-C(4)-C(3)	112.2(4)	H(11A)-C(11)-H(11C)	109.5
N(2)-C(5)-H(5A)	109.5	H(11B)-C(11)-H(11C)	109.5
N(2)-C(5)-H(5B)	109.5	N(4)-C(12)-H(12A)	109.5
H(5A)-C(5)-H(5B)	109.5	N(4)-C(12)-H(12B)	109.5
N(2)-C(5)-H(5C)	109.5	H(12A)-C(12)-H(12B)	109.5
H(5A)-C(5)-H(5C)	109.5	N(4)-C(12)-H(12C)	109.5
H(5B)-C(5)-H(5C)	109.5	H(12A)-C(12)-H(12C)	109.5
N(3)-C(6)-C(7)	125.1(5)	H(12B)-C(12)-H(12C)	109.5
N(3)-C(6)-H(6)	117.4	O(3)-C(13)-Re(1)	178.7(5)
C(7)-C(6)-H(6)	117.4	O(4)-C(14)-Re(1)	179.2(5)
C(6)-C(7)-C(8)	120.1(5)	O(5)-C(15)-Re(1)	176.3(5)
C(6)-C(7)-H(7)	119.9		

Symmetry transformations used to generate equivalent atoms:

Table 17. Crystal data and structure refinement for **6c**.

Identification code	**6c**	
Empirical formula	C21 H22 N5 O6 Re	
Formula weight	626.63	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.0901(17) Å	a= 101.832(2)°.
	b = 9.3897(18) Å	b= 98.774(2)°.
	c = 14.340(3) Å	g = 94.619(2)°.
Volume	1176.0(4) $Å^3$	
Z	2	
Density (calculated)	1.770 Mg/m^3	
Absorption coefficient	5.212 mm^{-1}	
F(000)	612	
Crystal size	0.450 x 0.450 x 0.400 mm^3	
Theta range for data collection	2.231 to 27.506°.	
Index ranges	-11<=h<=11, -12<=k<=12, -17<=l<=18	
Reflections collected	22902	
Independent reflections	5324 [R(int) = 0.0369]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.23 and 0.12	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5324 / 0 / 305	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0166, wR2 = 0.0429	
R indices (all data)	R1 = 0.0177, wR2 = 0.0432	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.290 and -1.186 $e.Å^{-3}$	

Table 18. Bond lengths [Å] and angles [°] for **6c**.

Re(1)-C(18)	1.903(2)	C(1M)-H(1M2)	0.9800
Re(1)-C(19)	1.919(3)	C(1M)-H(1M3)	0.9800
Re(1)-C(20)	1.923(2)	C(7)-C(8)	1.388(4)
Re(1)-N(1)	2.153(2)	C(7)-H(7)	0.9500
Re(1)-O(1)	2.1613(16)	C(8)-C(9)	1.378(4)
Re(1)-N(4)	2.1905(19)	C(8)-H(8)	0.9500
O(1)-C(3)	1.278(3)	C(9)-C(10)	1.395(4)
N(1)-N(3)	1.320(3)	C(9)-H(9)	0.9500
N(1)-C(1)	1.357(3)	C(10)-H(10)	0.9500
C(1)-C(2)	1.370(3)	C(11)-C(12)	1.370(3)
C(1)-C(3)	1.490(3)	C(11)-H(11)	0.9500
O(2)-C(3)	1.235(3)	C(12)-C(13)	1.417(3)
N(2)-C(2)	1.344(3)	C(12)-H(12)	0.9500
N(2)-N(3)	1.346(3)	C(13)-C(14)	1.418(3)
N(2)-C(4)	1.474(3)	C(14)-C(15)	1.368(3)
C(2)-H(2)	0.9500	C(14)-H(14)	0.9500
O(3)-C(18)	1.159(3)	C(15)-H(15)	0.9500
O(4)-C(19)	1.153(3)	C(16)-H(16A)	0.9800
N(4)-C(15)	1.354(3)	C(16)-H(16B)	0.9800
N(4)-C(11)	1.359(3)	C(16)-H(16C)	0.9800
C(4)-C(5)	1.510(3)	C(17)-H(17A)	0.9800
C(4)-H(4A)	0.9900	C(17)-H(17B)	0.9800
C(4)-H(4B)	0.9900	C(17)-H(17C)	0.9800
O(5)-C(20)	1.154(3)		
N(5)-C(13)	1.345(3)	C(18)-Re(1)-C(19)	90.72(10)
N(5)-C(17)	1.458(3)	C(18)-Re(1)-C(20)	90.56(10)
N(5)-C(16)	1.463(3)	C(19)-Re(1)-C(20)	89.09(10)
C(5)-C(6)	1.385(4)	C(18)-Re(1)-N(1)	98.34(9)
C(5)-C(10)	1.390(4)	C(19)-Re(1)-N(1)	170.69(8)
O(1M)-C(1M)	1.411(3)	C(20)-Re(1)-N(1)	92.93(9)
O(1M)-H(1M)	0.83(5)	C(18)-Re(1)-O(1)	172.75(8)
C(6)-C(7)	1.387(4)	C(19)-Re(1)-O(1)	95.60(8)
C(6)-H(6)	0.9500	C(20)-Re(1)-O(1)	93.10(8)
C(1M)-H(1M1)	0.9800	N(1)-Re(1)-O(1)	75.23(7)

C(18)-Re(1)-N(4)	94.95(8)	C(6)-C(5)-C(4)	120.5(2)
C(19)-Re(1)-N(4)	95.31(9)	C(10)-C(5)-C(4)	120.2(2)
C(20)-Re(1)-N(4)	172.90(8)	C(1M)-O(1M)-H(1M)	111(3)
N(1)-Re(1)-N(4)	81.86(7)	C(5)-C(6)-C(7)	120.3(3)
O(1)-Re(1)-N(4)	80.95(7)	C(5)-C(6)-H(6)	119.8
C(3)-O(1)-Re(1)	118.86(14)	C(7)-C(6)-H(6)	119.8
N(3)-N(1)-C(1)	110.52(19)	O(1M)-C(1M)-H(1M1)	109.5
N(3)-N(1)-Re(1)	134.98(15)	O(1M)-C(1M)-H(1M2)	109.5
C(1)-N(1)-Re(1)	114.50(15)	H(1M1)-C(1M)-H(1M2)	109.5
N(1)-C(1)-C(2)	107.6(2)	O(1M)-C(1M)-H(1M3)	109.5
N(1)-C(1)-C(3)	117.1(2)	H(1M1)-C(1M)-H(1M3)	109.5
C(2)-C(1)-C(3)	135.3(2)	H(1M2)-C(1M)-H(1M3)	109.5
C(2)-N(2)-N(3)	112.5(2)	C(6)-C(7)-C(8)	120.5(3)
C(2)-N(2)-C(4)	127.8(2)	C(6)-C(7)-H(7)	119.8
N(3)-N(2)-C(4)	119.67(19)	C(8)-C(7)-H(7)	119.8
N(2)-C(2)-C(1)	104.3(2)	C(9)-C(8)-C(7)	119.4(3)
N(2)-C(2)-H(2)	127.8	C(9)-C(8)-H(8)	120.3
C(1)-C(2)-H(2)	127.8	C(7)-C(8)-H(8)	120.3
N(1)-N(3)-N(2)	104.96(18)	C(8)-C(9)-C(10)	120.3(3)
O(2)-C(3)-O(1)	126.0(2)	C(8)-C(9)-H(9)	119.9
O(2)-C(3)-C(1)	119.8(2)	C(10)-C(9)-H(9)	119.9
O(1)-C(3)-C(1)	114.17(19)	C(5)-C(10)-C(9)	120.2(3)
C(15)-N(4)-C(11)	116.27(19)	C(5)-C(10)-H(10)	119.9
C(15)-N(4)-Re(1)	122.15(15)	C(9)-C(10)-H(10)	119.9
C(11)-N(4)-Re(1)	121.06(15)	N(4)-C(11)-C(12)	123.9(2)
N(2)-C(4)-C(5)	110.80(19)	N(4)-C(11)-H(11)	118.0
N(2)-C(4)-H(4A)	109.5	C(12)-C(11)-H(11)	118.0
C(5)-C(4)-H(4A)	109.5	C(11)-C(12)-C(13)	120.2(2)
N(2)-C(4)-H(4B)	109.5	C(11)-C(12)-H(12)	119.9
C(5)-C(4)-H(4B)	109.5	C(13)-C(12)-H(12)	119.9
H(4A)-C(4)-H(4B)	108.1	N(5)-C(13)-C(12)	122.2(2)
C(13)-N(5)-C(17)	120.1(2)	N(5)-C(13)-C(14)	122.5(2)
C(13)-N(5)-C(16)	121.0(2)	C(12)-C(13)-C(14)	115.3(2)
C(17)-N(5)-C(16)	118.8(2)	C(15)-C(14)-C(13)	120.7(2)
C(6)-C(5)-C(10)	119.2(2)	C(15)-C(14)-H(14)	119.6

C(13)-C(14)-H(14)	119.6	N(5)-C(17)-H(17A)	109.5
N(4)-C(15)-C(14)	123.6(2)	N(5)-C(17)-H(17B)	109.5
N(4)-C(15)-H(15)	118.2	H(17A)-C(17)-H(17B)	109.5
C(14)-C(15)-H(15)	118.2	N(5)-C(17)-H(17C)	109.5
N(5)-C(16)-H(16A)	109.5	H(17A)-C(17)-H(17C)	109.5
N(5)-C(16)-H(16B)	109.5	H(17B)-C(17)-H(17C)	109.5
H(16A)-C(16)-H(16B)	109.5	O(3)-C(18)-Re(1)	177.5(2)
N(5)-C(16)-H(16C)	109.5	O(4)-C(19)-Re(1)	178.3(2)
H(16A)-C(16)-H(16C)	109.5	O(5)-C(20)-Re(1)	177.9(2)
H(16B)-C(16)-H(16C)	109.5		

Symmetry transformations used to generate equivalent atoms: