Crystallographic Data of the Thesis

"Complexes of Rhenium and Technetium with Chelating Thiourea Ligands"

Submitted by

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1 H_2picbtu

Table 1.1 Crystal data and structure refinement for H_2picbtu.

Empirical formula	$C_{14}H_{13}N_3OS$	
Formula weight	271.34	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 8.492(1) Å	α= 97.75(1)°
	b = 8.759(1) Å	β= 96.04(1)°
	c = 8.958(1) Å	γ = 90.84(1)°
Volume	656.3(1) $Å^3$	
Z	2	
Density (calculated)	1.373 g/cm^3	
Absorption coefficient	0.241 mm^{-1}	
F(000)	284	
Crystal description	Block	
Crystal color	Colorless	
Crystal size	0.5 x 0.4 x 0.3 mm^3	
Theta range for data collection	2.31 to 29.26	
Index ranges	-9<=h<=11, -11<=k<=12, -12<=l<=12	
Reflections collected	6812	
Independent reflections	3489 [R(int) = 0.0647]	
Completeness to theta = 29.26°	97.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3489 / 0 / 173	
Goodness-of-fit on F^2	0.888	
Final R indices [I>2sigma(I)]	R1 = 0.0390, wR2 = 0.0930	
R indices (all data)	R1 = 0.0622, wR2 = 0.0990	
Extinction coefficient	0.043(7)	
Largest diff. peak and hole	0.281 and -0.244 $e.Å^{-3}$	

Table 1.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for H_2picbtu. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	464(1)	271(1)	2358(1)	39(1)
C(2)	-1019(2)	1502(2)	2207(1)	29(1)
N(3)	-1934(2)	1471(1)	819(1)	32(1)
C(4)	-3241(2)	2329(2)	505(2)	32(1)
O(5)	-3602(1)	3420(1)	1392(1)	43(1)
N(6)	-1392(2)	2495(1)	3344(1)	32(1)
C(7)	-581(2)	2611(2)	4860(2)	35(1)
C(41)	-1346(2)	3793(2)	5924(2)	32(1)
C(42)	-708(2)	4129(2)	7407(2)	57(1)
C(43)	-1415(3)	5235(3)	8352(2)	64(1)
C(44)	-2726(2)	5931(2)	7807(2)	43(1)
C(45)	-3296(2)	5517(2)	6327(2)	49(1)
N(46)	-2623(2)	4463(2)	5375(1)	45(1)
C(31)	-4232(2)	1848(2)	-962(2)	30(1)
C(32)	-4295(2)	344(2)	-1711(2)	34(1)
C(33)	-5323(2)	-47(2)	-3015(2)	41(1)
C(34)	-6286(2)	1040(2)	-3583(2)	45(1)
C(35)	-6222(2)	2541(2)	-2847(2)	44(1)
C(36)	-5213(2)	2938(2)	-1539(2)	37(1)

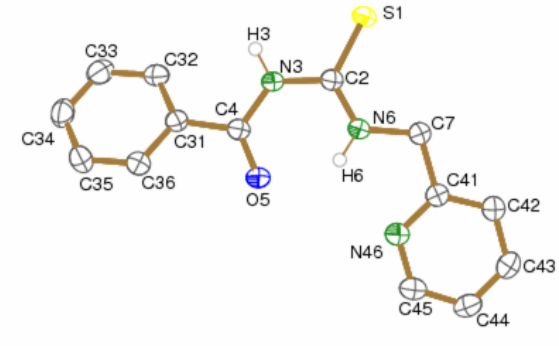

Figure 1 Ellipsoid plot (50% probability) of H_2picbtu.

2 [$ReOCl_2(PPh_3)(i\text{-}Pr_2btu)$], (1d)

Table 2.1 Crystal data and structure refinement for [$ReOCl_2(PPh_3)(i\text{-}Pr_2btu)$].

Empirical formula	$C_{32}H_{34}Cl_2N_2O_2PReS$	
Formula weight	798.74	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.911(1) Å	α= 90°
	b = 19.233(1) Å	β= 92.02(1)°
	c = 16.768(1) Å	γ = 90°
Volume	3194.3(4) $Å^3$	
Z	4	
Density (calculated)	1.661 g/cm^3	
Absorption coefficient	4.119 mm^{-1}	
F(000)	1584	
Crystal description	Block	
Crystal color	Green	
Crystal size	0.320 x 0.177 x 0.090 mm^3	
Theta range for data collection	2.95 to 29.33	
Index ranges	-13<=h<=12, -26<=k<=25, -22<=l<=22	
Reflections collected	22445	
Independent reflections	8558 [R(int) = 0.0516]	
Completeness to theta = 29.33°	97.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.6600 and 0.4327	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8558 / 0 / 370	
Goodness-of-fit on F^2	0.913	
Final R indices [I>2sigma(I)]	R1 = 0.0440, wR2 = 0.1009	
R indices (all data)	R1 = 0.0685, wR2 = 0.1102	
Largest diff. peak and hole	1.057 and -2.811 $e.Å^{-3}$	

Table 2.1 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$ReOCl_2(PPh_3)$(*i*-Pr_2btu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	5294(1)	4190(1)	8020(1)	29(1)
O(10)	4126(4)	4384(2)	8686(2)	43(1)
Cl(2)	5812(2)	3036(1)	8489(1)	41(1)
Cl(1)	3958(1)	3650(1)	6987(1)	43(1)
S(1)	7179(1)	4646(1)	8782(1)	37(1)
C(2)	8362(5)	3967(3)	8702(3)	33(1)
N(3)	8788(4)	3763(2)	7982(3)	34(1)
C(4)	8070(5)	3843(3)	7315(3)	29(1)
O(5)	6815(4)	4013(2)	7232(2)	32(1)
C(31)	8758(5)	3685(3)	6564(3)	32(1)
C(32)	8081(6)	3778(3)	5831(3)	41(1)
C(33)	8718(7)	3610(3)	5128(4)	46(1)
C(34)	10002(7)	3345(3)	5151(4)	47(2)
C(35)	10696(6)	3258(3)	5877(4)	46(1)
C(36)	10086(6)	3433(3)	6580(3)	39(1)
N(6)	9000(4)	3717(2)	9348(3)	33(1)
C(44)	10180(6)	3242(3)	9291(3)	41(1)
C(46)	9827(9)	2556(4)	8886(5)	65(2)
C(45)	11396(6)	3610(4)	8952(4)	59(2)
C(41)	8516(7)	3842(4)	10163(3)	49(2)
C(43)	8008(8)	3174(5)	10515(4)	64(2)
C(42)	9574(11)	4198(4)	10693(5)	72(2)
P(1)	5005(1)	5370(1)	7456(1)	30(1)
C(51)	5771(6)	6026(3)	8112(3)	35(1)
C(52)	5386(7)	6025(3)	8909(4)	42(1)
C(53)	5964(8)	6498(4)	9444(4)	53(2)
C(54)	6936(8)	6962(4)	9190(4)	55(2)
C(55)	7300(7)	6973(3)	8406(4)	51(2)
C(56)	6717(6)	6505(3)	7866(4)	40(1)
C(61)	3254(5)	5664(3)	7330(3)	34(1)
C(62)	2208(6)	5200(3)	7202(4)	44(1)
C(63)	900(7)	5450(4)	7073(4)	53(2)
C(64)	642(6)	6151(4)	7084(4)	50(2)
C(65)	1672(6)	6613(4)	7216(3)	45(1)

C(66)	2988(6)	6374(3)	7340(3)	41(1)
C(71)	5758(5)	5503(3)	6496(3)	31(1)
C(72)	7150(5)	5411(3)	6434(3)	36(1)
C(73)	7737(6)	5512(3)	5709(4)	43(1)
C(74)	6953(6)	5705(3)	5044(3)	42(1)
C(75)	5591(7)	5804(3)	5104(4)	45(1)
C(76)	4975(6)	5702(3)	5829(3)	38(1)

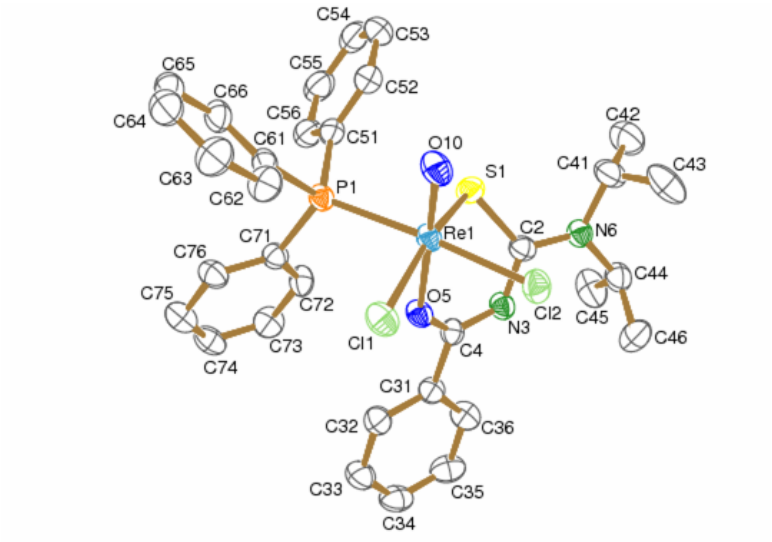

Figure 2 Ellipsoid plot (50% probability) of [$ReOCl_2(PPh_3)$(*i*-Pr_2btu)].

3 [$ReOCl_2(PPh_3)$(Morphbtu)], (1e)

Table 3.1 Crystal data and structure refinement for [$ReOCl_2(PPh_3)$(Morphbtu)].

Empirical formula	$C_{30}H_{28}Cl_2N_2O_3PReS$	
Formula weight	784.47	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.730(1) Å	α= 90°
	b = 19.891(1) Å	β= 130.80(1)°
	c = 16.864(2) Å	γ = 90°
Volume	2978.7(5) $Å^3$	
Z	4	
Density (calculated)	1.749 g/cm^3	
Absorption coefficient	4.418 mm^{-1}	
F(000)	1544	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.160 x 0.124 x 0.105 mm^3	
Theta range for data collection	2.69 to 29.24	
Index ranges	-16<=h<=15, -27<=k<=27, -17<=l<=23	
Reflections collected	21879	
Independent reflections	8007 [R(int) = 0.0309]	
Completeness to theta = 29.24°	98.8 %	
Absorption correction	Integration	
Max. and min. transmission	0.6017 and 0.2067	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8007 / 0 / 361	
Goodness-of-fit on F^2	1.042	
Final R indices [I>2sigma(I)]	R1 = 0.0284, wR2 = 0.0644	
R indices (all data)	R1 = 0.0366, wR2 = 0.0667	
Largest diff. peak and hole	0.859 and -2.802 e.$Å^{-3}$	

Table 3.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$ReOCl_2(PPh_3)$(Morphbtu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	1903(1)	-1321(1)	5338(1)	20(1)
Cl(2)	322(1)	-1759(1)	3586(1)	29(1)
Cl(1)	2268(1)	-2446(1)	5942(1)	33(1)
O(10)	527(2)	-1063(1)	5333(2)	30(1)
S(1)	2112(1)	-273(1)	4735(1)	28(1)
C(2)	2094(3)	-574(1)	3745(3)	26(1)
N(3)	3203(3)	-969(1)	3974(2)	27(1)
C(4)	3964(3)	-1381(1)	4778(2)	24(1)
O(5)	3598(2)	-1567(1)	5319(2)	24(1)
C(31)	5376(3)	-1680(1)	5109(2)	24(1)
C(32)	5943(3)	-2258(2)	5717(3)	29(1)
C(33)	7311(4)	-2520(2)	6087(3)	34(1)
C(34)	8131(4)	-2190(2)	5883(3)	36(1)
C(35)	7578(4)	-1610(2)	5284(3)	38(1)
C(36)	6180(4)	-1362(2)	4871(3)	32(1)
N(6)	1099(3)	-343(1)	2777(2)	27(1)
C(41)	-248(4)	51(2)	2379(3)	35(1)
C(42)	-1634(4)	-337(2)	1520(4)	47(1)
O(43)	-1631(3)	-502(2)	700(2)	58(1)
C(44)	-364(5)	-910(2)	1100(3)	48(1)
C(45)	1092(4)	-553(2)	1940(3)	34(1)
P	3887(1)	-949(1)	7163(1)	21(1)
C(51)	3585(3)	-1189(1)	8055(2)	23(1)
C(52)	2346(4)	-1570(2)	7723(3)	33(1)
C(53)	2145(4)	-1728(2)	8430(3)	38(1)
C(54)	3155(4)	-1502(2)	9450(3)	39(1)
C(55)	4380(4)	-1117(2)	9778(3)	39(1)
C(56)	4601(3)	-961(2)	9091(2)	31(1)
C(61)	5714(3)	-1273(1)	7705(2)	24(1)
C(62)	6401(3)	-1005(2)	7340(3)	27(1)
C(63)	7779(4)	-1245(2)	7718(3)	35(1)
C(64)	8487(4)	-1753(2)	8458(3)	39(1)
C(65)	7805(4)	-2028(2)	8805(3)	40(1)
C(66)	6412(4)	-1794(2)	8432(3)	32(1)

C(71)	4135(3)	-40(1)	7341(2)	22(1)
C(72)	5558(3)	260(1)	7996(3)	28(1)
C(73)	5696(4)	955(2)	8113(3)	32(1)
C(74)	4429(4)	1352(2)	7605(3)	33(1)
C(75)	3018(4)	1059(2)	6978(3)	33(1)
C(76)	2861(3)	365(2)	6833(3)	27(1)

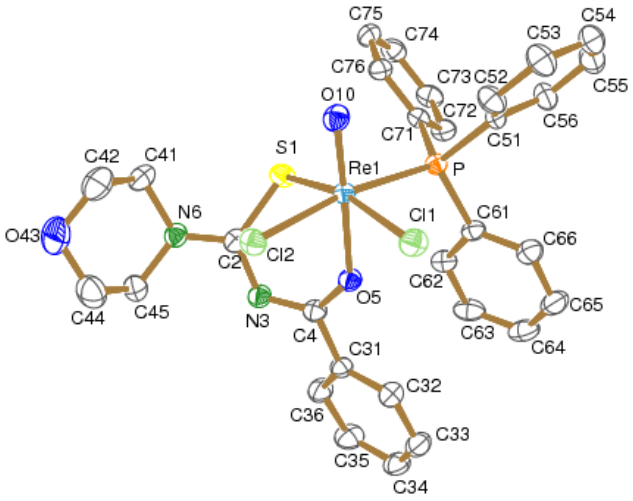

Figure 3 Ellipsoid plot (50% probability) of [$ReOCl_2(PPh_3)$(Morphbtu)].

4 [ReOCl(PhMebtu)$_2$], (2b)

Table 4.1 Crystal data and structure refinement for [ReOCl(PhMebtu)$_2$].

Empirical formula	$C_{30}H_{26}ClN_4O_3ReS_2$	
Formula weight	776.32	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 19.727(1) Å	α= 90°
	b = 18.811(1) Å	β= 117.85(1)°
	c = 18.511(1) Å	γ = 90°
Volume	6073.7(6) Å^3	
Z	8	
Density (calculated)	1.698 g/cm^3	
Absorption coefficient	4.265 mm^{-1}	
F(000)	3056	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.200 x 0.173 x 0.150 mm^3	
Theta range for data collection	1.65 to 29.32	
Index ranges	-27<=h<=27, -22<=k<=25, -25<=l<=25	
Reflections collected	36916	
Independent reflections	16246 [R(int) = 0.0896]	
Completeness to theta = 29.32°	97.6 %	
Absorption correction	Integration	
Max. and min. transmission	0.6203 and 0.5013	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	16246 / 0 / 739	
Goodness-of-fit on F^2	0.946	
Final R indices [I>2sigma(I)]	R1 = 0.0489, wR2 = 0.0975	
R indices (all data)	R1 = 0.1072, wR2 = 0.1263	
Largest diff. peak and hole	1.359 and -1.896 e.Å^{-3}	

Table 4.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReOCl(PhMebtu)$_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	4345(1)	-1899(1)	1841(1)	28(1)
Cl(1)	3694(1)	-1285(1)	537(1)	40(1)
O(10)	4556(3)	-2565(3)	1385(4)	41(1)
S(11)	4870(1)	-2314(1)	3173(1)	33(1)
C(12)	5859(4)	-2120(4)	3619(5)	27(2)
N(13)	6171(4)	-1552(4)	3433(5)	36(2)
C(14)	5868(4)	-1162(4)	2769(5)	27(2)
O(15)	5278(3)	-1270(3)	2084(4)	34(1)
C(51)	6284(5)	-490(4)	2812(5)	34(2)
C(52)	7035(5)	-389(5)	3426(6)	47(2)
C(53)	7406(7)	241(6)	3435(9)	71(4)
C(54)	7052(8)	767(6)	2890(9)	68(4)
C(55)	6315(8)	667(6)	2273(9)	70(3)
C(56)	5934(6)	46(5)	2242(7)	53(3)
N(16)	6323(3)	-2539(4)	4205(4)	32(1)
C(61)	6048(4)	-3107(4)	4536(5)	33(2)
C(62)	5703(5)	-2943(5)	5006(6)	42(2)
C(63)	5472(6)	-3478(6)	5347(7)	54(3)
C(64)	5590(6)	-4174(6)	5215(7)	55(3)
C(65)	5912(6)	-4348(5)	4713(7)	51(2)
C(66)	6161(5)	-3797(5)	4392(6)	40(2)
C(67)	7168(5)	-2434(6)	4586(6)	47(2)
S(1)	3179(1)	-2281(1)	1754(2)	36(1)
C(2)	2549(4)	-1555(4)	1344(5)	30(2)
N(3)	2709(3)	-869(3)	1506(4)	28(1)
C(4)	3412(4)	-619(4)	1948(5)	30(2)
O(5)	4052(3)	-958(3)	2236(4)	32(1)
C(31)	3494(5)	151(4)	2149(5)	31(2)
C(32)	4222(5)	443(4)	2603(6)	40(2)
C(33)	4303(7)	1159(5)	2793(7)	57(3)
C(34)	3668(6)	1591(5)	2519(6)	49(2)
C(35)	2944(6)	1313(5)	2077(7)	49(3)
C(36)	2860(5)	592(4)	1899(6)	37(2)
N(6)	1817(3)	-1728(3)	901(4)	31(2)

C(47)	1207(5)	-1183(5)	665(8)	57(3)
C(41)	1551(4)	-2440(4)	596(5)	29(2)
C(46)	1180(5)	-2822(5)	937(6)	41(2)
C(45)	881(5)	-3483(5)	634(6)	43(2)
C(44)	984(5)	-3770(5)	15(6)	43(2)
C(43)	1354(7)	-3394(6)	-321(7)	60(3)
C(42)	1643(6)	-2722(5)	-43(6)	47(2)
Re(11)	690(1)	-5370(1)	7969(1)	28(1)
Cl(11)	1304(1)	-5883(1)	9326(1)	44(1)
O(20)	481(3)	-4644(3)	8334(4)	45(2)
S(111)	155(1)	-5062(1)	6586(1)	31(1)
C(112)	-822(4)	-5176(4)	6271(5)	31(2)
N(113)	-1137(4)	-5727(4)	6451(4)	37(2)
C(114)	-819(4)	-6135(4)	7086(5)	28(2)
O(115)	-214(3)	-6023(3)	7767(4)	33(1)
C(151)	-1219(5)	-6814(4)	7069(5)	31(2)
C(152)	-2008(5)	-6825(5)	6708(6)	42(2)
C(153)	-2387(6)	-7449(5)	6685(7)	51(3)
C(154)	-1984(7)	-8059(5)	7010(8)	61(3)
C(155)	-1186(6)	-8058(4)	7360(6)	43(2)
C(156)	-804(5)	-7426(4)	7401(6)	38(2)
N(116)	-1309(4)	-4707(4)	5751(4)	35(2)
C(161)	-1069(4)	-4075(4)	5488(4)	26(2)
C(162)	-1040(5)	-3435(5)	5844(6)	42(2)
C(163)	-879(6)	-2817(5)	5528(7)	52(3)
C(164)	-772(5)	-2855(5)	4870(7)	48(2)
C(165)	-808(5)	-3507(5)	4482(6)	43(2)
C(166)	-946(4)	-4115(5)	4802(5)	35(2)
C(167)	-2147(4)	-4788(5)	5438(6)	47(2)
S(101)	1851(1)	-5035(1)	8006(2)	37(1)
C(102)	2477(4)	-5707(4)	8605(5)	31(2)
C(104)	1685(4)	-6673(4)	7976(5)	26(2)
N(103)	2361(4)	-6404(3)	8487(5)	35(2)
O(105)	1030(3)	-6368(3)	7690(4)	33(1)
C(131)	1677(4)	-7435(4)	7746(5)	30(2)
C(132)	1000(5)	-7756(4)	7193(6)	38(2)
C(133)	991(6)	-8464(5)	6987(7)	53(3)
C(134)	1663(7)	-8866(5)	7335(7)	59(3)
C(135)	2336(6)	-8547(6)	7888(8)	61(3)

C(136)	2349(5)	-7839(5)	8103(6)	44(2)
N(106)	3178(4)	-5500(3)	9181(5)	36(2)
C(147)	3783(5)	-6023(5)	9620(7)	56(3)
C(141)	3375(5)	-4772(5)	9378(6)	38(2)
C(142)	3165(6)	-4422(5)	9892(6)	50(2)
C(143)	3404(6)	-3735(6)	10141(7)	59(3)
C(144)	3836(6)	-3391(6)	9839(7)	57(3)
C(145)	4031(6)	-3719(5)	9303(6)	49(2)
C(146)	3813(5)	-4424(5)	9083(6)	41(2)

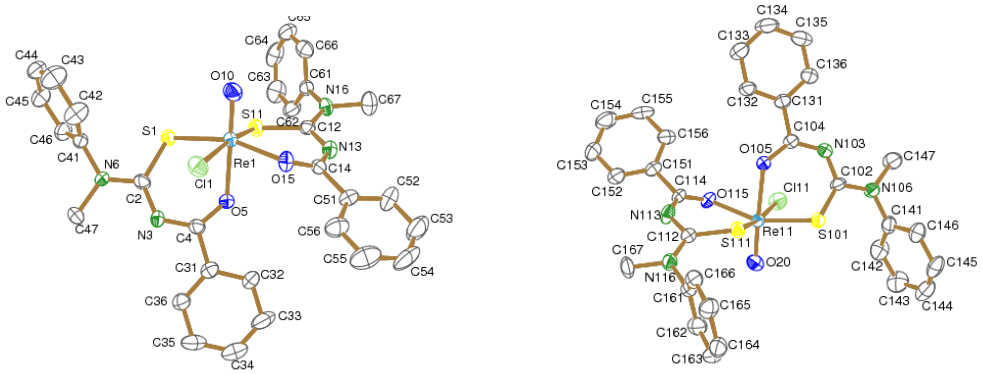

Figure 4 Ellipsoid plot (50% probability) of two crystallographically independent species of $[ReOCl(PhMebtu)_2]$.

5 [ReO(OMe)(Et_2btu)$_2$], (3c)

Table 5.1 Crystal data and structure refinement for [ReO(OMe)(Et_2btu)$_2$].

Empirical formula	$C_{25}H_{33}N_4O_4ReS_2$	
Formula weight	703.90	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 31.573(2) Å	α= 90°
	b = 11.338(1) Å	β= 109.85(1)°
	c = 16.041(1) Å	γ = 90°
Volume	5400.9(7) Å^3	
Z	8	
Density (calculated)	1.731 g/cm^3	
Absorption coefficient	4.693 mm^{-1}	
F(000)	2799	
Crystal description	Block	
Crystal color	Violet	
Crystal size	0.56 x 0.1 x 0.06 mm^3	
Theta range for data collection	1.92 to 26.83	
Index ranges	-33<=h<=39, -14<=k<=14, -20<=l<=20	
Reflections collected	23279	
Independent reflections	5740 [R(int) = 0.0890]	
Completeness to theta = 26.83°	98.8 %	
Absorption correction	Integration	
Max. and min. transmission	0.5844 and 0.3657	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5740 / 0 / 344	
Goodness-of-fit on F^2	0.950	
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.0730	
R indices (all data)	R1 = 0.0654, wR2 = 0.0786	
Largest diff. peak and hole	3.211 and -1.755 e.Å^{-3}	

Table 5.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(OMe)(Et_2btu)$_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	1205(1)	2323(1)	1194(1)	33(1)
O(10)	1397(1)	2497(4)	2312(4)	57(1)
O(20)	1071(1)	2570(3)	27(4)	67(2)
C(20)	1115(3)	3503(6)	-587(6)	61(2)
S(11)	510(1)	1493(1)	1029(1)	37(1)
C(12)	123(2)	2647(5)	924(4)	30(1)
N(13)	210(2)	3806(4)	1093(3)	29(1)
C(14)	577(2)	4390(4)	1196(4)	28(1)
O(15)	945(1)	4067(3)	1115(4)	47(1)
C(51)	561(2)	5673(4)	1397(4)	29(1)
C(52)	156(2)	6222(5)	1304(5)	49(2)
C(53)	145(3)	7414(6)	1491(7)	70(2)
C(54)	536(3)	8054(5)	1767(5)	57(2)
C(55)	937(3)	7532(5)	1860(5)	54(2)
C(56)	952(2)	6332(5)	1669(5)	42(2)
N(16)	-309(1)	2342(4)	703(3)	32(1)
C(63)	-652(2)	3231(5)	693(4)	37(1)
C(64)	-665(2)	3500(6)	1604(5)	49(2)
C(61)	-473(2)	1124(5)	467(5)	41(2)
C(62)	-510(3)	419(6)	1228(6)	60(2)
S(1)	1498(1)	456(1)	1129(1)	34(1)
C(2)	2065(2)	632(5)	1244(4)	37(1)
N(3)	2297(2)	1614(4)	1211(4)	41(1)
C(4)	2172(2)	2722(5)	1190(4)	35(1)
O(5)	1811(1)	3172(3)	1216(4)	54(1)
C(31)	2529(2)	3585(5)	1186(4)	31(1)
C(32)	2483(2)	4771(5)	1381(4)	39(1)
C(33)	2827(2)	5563(5)	1449(5)	41(2)
C(34)	3211(2)	5192(5)	1308(5)	43(2)
C(35)	3252(2)	4033(5)	1076(5)	44(2)
C(36)	2917(2)	3236(5)	1032(4)	36(1)
N(6)	2310(2)	-365(4)	1384(5)	60(2)
C(41)	2131(2)	-1516(5)	1492(5)	51(2)
C(42)	1905(2)	-2151(6)	636(5)	55(2)

C(43A)	2813(5)	-335(11)	1764(11)	42(5)
C(44A)	2965(6)	-574(16)	1006(12)	65(5)
C(43B)	2726(6)	-329(11)	1050(12)	37(5)
C(44B)	3119(6)	18(12)	1879(10)	43(5)

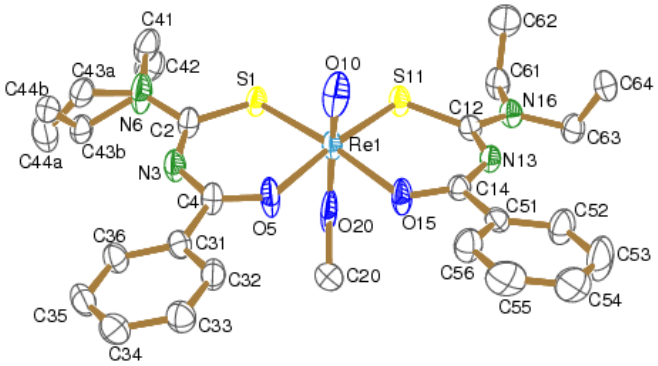

Figure 5 Ellipsoid plot (50% probability) of [ReO(OMe)(Et_2btu)$_2$].

6 [{ReO(Et$_2$btu)$_2$}O], (4c)

Table 6.1 Crystal data and structure refinement for [{ReO(Et$_2$btu)$_2$}O].

Empirical formula	$C_{48}H_{60}N_8O_7Re_2S_4$	
Formula weight	1361.68	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 20.955(9) Å	α= 90°
	b = 15.933(9) Å	β= 108.50(5)°
	c = 16.661(12) Å	γ = 90°
Volume	5275(5) Å^3	
Z	4	
Density (calculated)	1.715 g/cm^3	
Absorption coefficient	4.800 mm^{-1}	
F(000)	2696	
Crystal description	Needle	
Crystal color	Green	
Crystal size	1.000 x 0.400 x 0.100 mm^3	
Theta range for data collection	1.88 to 29.28	
Index ranges	-23<=h<=28, -21<=k<=21, -22<=l<=22	
Reflections collected	56774	
Independent reflections	14195 [R(int) = 0.1978]	
Completeness to theta = 29.28°	98.6 %	
Absorption correction	Integration	
Max. and min. transmission	0.6845 and 0.4659	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14195 / 0 / 618	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0782, wR2 = 0.1123	
R indices (all data)	R1 = 0.1237, wR2 = 0.1225	
Extinction coefficient	0.00102(8)	
Largest diff. peak and hole	2.891 and -4.707 e.Å^{-3}	

Table 6.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(Et_2btu)$_2$}O]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	2175(1)	4602(1)	833(1)	27(1)
O(10)	1945(4)	3907(4)	1469(5)	47(2)
O(30)	2543(3)	5231(3)	119(4)	28(1)
S(1)	1102(1)	4684(1)	-169(2)	35(1)
C(2)	990(4)	3792(6)	-802(6)	32(2)
N(3)	1451(4)	3232(5)	-889(5)	37(2)
C(4)	2086(4)	3173(5)	-425(6)	26(2)
O(5)	2436(3)	3585(4)	210(5)	40(2)
C(31)	2453(4)	2446(5)	-653(6)	30(2)
C(32)	3096(5)	2250(5)	-119(6)	32(2)
C(33)	3423(5)	1528(6)	-246(8)	45(3)
C(34)	3099(6)	1011(6)	-929(8)	50(3)
C(35)	2484(6)	1221(7)	-1488(8)	56(3)
C(36)	2157(6)	1930(6)	-1343(7)	41(2)
N(6)	359(4)	3633(5)	-1282(6)	45(2)
C(43)	-217(5)	4148(7)	-1249(8)	48(3)
C(44)	-338(6)	4916(7)	-1820(7)	53(3)
C(41)	174(7)	2756(12)	-1763(9)	87(5)
C(42)	81(11)	2992(11)	-2610(15)	140(10)
S(11)	1989(1)	5821(1)	1504(2)	31(1)
C(12)	2696(4)	5955(5)	2418(5)	27(2)
N(13)	3318(3)	5667(4)	2561(5)	31(2)
C(14)	3523(4)	5070(5)	2147(6)	29(2)
O(15)	3186(3)	4548(4)	1603(4)	37(2)
C(51)	4267(4)	4955(5)	2395(6)	31(2)
C(52)	4543(5)	4536(7)	1863(8)	48(3)
C(53)	5246(6)	4427(7)	2103(9)	64(4)
C(54)	5652(5)	4743(7)	2832(9)	64(4)
C(55)	5388(5)	5183(7)	3366(9)	60(3)
C(56)	4693(5)	5286(6)	3150(7)	47(3)
N(16)	2606(4)	6434(4)	3024(5)	31(2)
C(61)	1949(5)	6783(6)	2983(7)	42(3)
C(62)	1793(6)	7603(6)	2529(9)	63(4)
C(63)	3168(5)	6669(6)	3759(6)	36(2)

C(64)	3280(5)	6035(7)	4483(7)	49(3)
Re(11)	2857(1)	5954(1)	-593(1)	30(1)
O(20)	2951(5)	6623(4)	-1333(5)	58(2)
S(101)	3725(1)	5029(2)	-552(2)	41(1)
C(102)	3407(5)	4246(5)	-1304(7)	36(2)
N(103)	2782(4)	4157(4)	-1856(5)	33(2)
C(104)	2252(4)	4622(6)	-1946(5)	31(2)
O(105)	2153(3)	5264(4)	-1555(4)	42(2)
C(131)	1647(4)	4396(5)	-2697(6)	31(2)
C(132)	1064(5)	4874(6)	-2916(7)	39(2)
C(133)	535(5)	4710(7)	-3640(7)	45(2)
C(134)	573(5)	4073(8)	-4167(7)	55(3)
C(135)	1144(6)	3571(7)	-3946(7)	49(3)
C(136)	1672(5)	3726(6)	-3213(7)	43(3)
N(106)	3840(4)	3662(5)	-1358(5)	35(2)
C(141)	3614(5)	2930(6)	-1931(7)	42(2)
C(142)	3627(7)	3097(8)	-2816(8)	60(3)
C(143)	4562(5)	3695(6)	-900(7)	38(2)
C(144)	4759(6)	3187(8)	-87(8)	55(3)
S(111)	3573(1)	6633(1)	594(2)	37(1)
C(112)	3138(6)	7498(6)	795(7)	44(3)
N(113)	2481(4)	7664(5)	535(6)	41(2)
C(114)	1993(5)	7251(5)	-26(6)	35(2)
O(115)	2021(3)	6671(4)	-541(5)	46(2)
C(151)	1299(6)	7510(7)	-79(7)	44(3)
C(152)	1191(7)	8275(8)	273(8)	60(3)
C(153)	545(9)	8476(11)	250(10)	92(6)
C(154)	29(8)	7978(15)	-107(10)	97(7)
C(155)	128(7)	7211(12)	-450(9)	80(5)
C(156)	772(5)	6990(8)	-465(8)	51(3)
N(116)	3504(5)	8067(7)	1336(9)	85(5)
C(163)	3205(7)	8846(10)	1554(12)	98(7)
C(164)	3188(10)	9533(11)	963(16)	150(11)
C(161)	4381(16)	8137(11)	1649(14)	180(15)
C(162)	4351(9)	7682(9)	2287(14)	105(7)

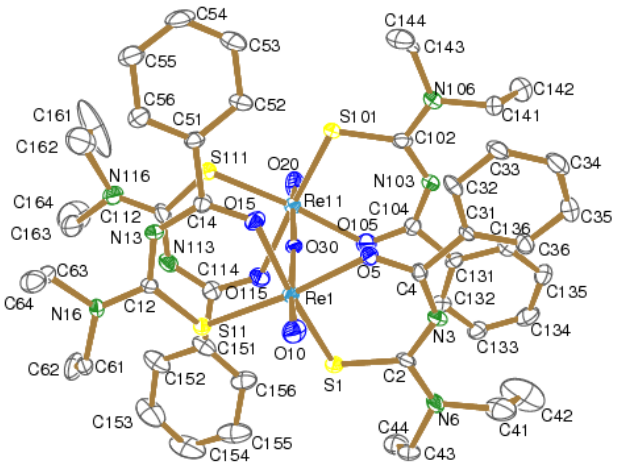

Figure 6 Ellipsoid plot (50% probability) of [{ReO(Et_2btu)$_2$}O].

7 [TcOCl(Ph$_2$btu)$_2$], (5a)

Table 7.1 Crystal data and structure refinement for [TcOCl(Ph$_2$btu)$_2$].

Empirical formula	$C_{40}H_{30}ClN_4O_3S_2Tc$	
Formula weight	812.25	
Temperature	200(2) K	
Wavelength	0.71069 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 10.763(1) Å	α= 92.48(1)°
	b = 11.207(1) Å	β= 99.99(1)°
	c = 16.926(1) Å	γ = 114.79(1)°
Volume	1809.9(3) Å^3	
Z	2	
Density (calculated)	1.490 g/cm^3	
Absorption coefficient	0.632 mm^{-1}	
F(000)	828	
Crystal description	Plate	
Crystal color	Yellow	
Crystal size	0.2 x 0.15 x 0.05 mm^3	
Theta range for data collection	2.02 to 29.31	
Index ranges	-14<=h<=14, -15<=k<=15, -23<=l<=23	
Reflections collected	33030	
Independent reflections	9807 [R(int) = 0.1540]	
Completeness to theta = 29.31°	98.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9807 / 0 / 460	
Goodness-of-fit on F^2	0.929	
Final R indices [I>2sigma(I)]	R1 = 0.0639, wR2 = 0.1104	
R indices (all data)	R1 = 0.1387, wR2 = 0.1474	
Largest diff. peak and hole	0.661 and -1.024 e.Å^{-3}	

Table 7.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$TcOCl(Ph_2btu)_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc(1)	5947(1)	6485(1)	6677(1)	25(1)
O(10)	5992(4)	5183(4)	6223(2)	37(1)
Cl(1)	5146(2)	5306(2)	7801(1)	38(1)
S(1)	8278(1)	7646(2)	7407(1)	32(1)
C(2)	8033(5)	7850(6)	8397(3)	32(1)
N(3)	7272(5)	8425(5)	8612(3)	33(1)
C(4)	6299(5)	8607(5)	8083(3)	30(1)
O(5)	5717(4)	7998(4)	7375(2)	29(1)
C(31)	5839(6)	9581(6)	8385(3)	36(1)
C(32)	4848(6)	9843(6)	7876(4)	40(1)
C(33)	4407(8)	10758(7)	8146(4)	54(2)
C(34)	4941(10)	11394(10)	8908(5)	83(3)
C(35)	5950(11)	11165(11)	9424(5)	105(4)
C(36)	6373(8)	10241(9)	9156(4)	73(3)
N(6)	8778(4)	7512(5)	9004(3)	32(1)
C(41)	8489(6)	7530(6)	9815(3)	35(1)
C(42)	9462(7)	8444(6)	10429(4)	42(1)
C(43)	9161(8)	8425(8)	11200(4)	54(2)
C(44)	7951(8)	7487(8)	11335(4)	57(2)
C(45)	6997(8)	6559(8)	10718(4)	55(2)
C(46)	7252(7)	6589(7)	9943(4)	47(2)
C(51)	9810(5)	7042(6)	8883(3)	33(1)
C(52)	9613(6)	5799(7)	9077(3)	41(1)
C(53)	10610(7)	5343(7)	8977(4)	50(2)
C(54)	11767(7)	6128(8)	8708(4)	52(2)
C(55)	11971(7)	7376(8)	8529(4)	56(2)
C(56)	10985(6)	7841(7)	8626(4)	43(1)
S(11)	6674(1)	7990(2)	5762(1)	31(1)
C(12)	5148(5)	7729(5)	5067(3)	29(1)
N(13)	3862(4)	7330(5)	5205(3)	32(1)
C(14)	3349(5)	6636(5)	5771(3)	27(1)
O(15)	3887(4)	6043(4)	6254(2)	32(1)
C(61)	1929(5)	6434(6)	5845(3)	33(1)
C(62)	1382(6)	5866(6)	6498(4)	42(1)

C(63)	47(7)	5619(8)	6559(4)	55(2)
C(64)	-763(6)	5924(7)	5963(5)	55(2)
C(65)	-272(6)	6473(7)	5321(4)	49(2)
C(66)	1084(5)	6742(6)	5250(4)	36(1)
N(16)	5325(4)	8095(4)	4328(2)	28(1)
C(71)	4126(5)	7990(6)	3718(3)	32(1)
C(72)	3469(6)	8781(6)	3830(4)	42(1)
C(73)	2360(6)	8683(7)	3236(4)	52(2)
C(74)	1919(7)	7826(7)	2551(5)	62(2)
C(75)	2613(7)	7054(7)	2435(4)	57(2)
C(76)	3731(6)	7140(6)	3025(4)	39(1)
C(81)	6652(5)	8527(5)	4087(3)	28(1)
C(82)	7137(6)	7626(6)	3881(3)	33(1)
C(83)	8376(6)	8061(7)	3605(4)	43(2)
C(84)	9079(7)	9375(7)	3535(4)	51(2)
C(85)	8575(7)	10268(7)	3714(4)	52(2)
C(86)	7341(6)	9828(6)	4001(4)	43(1)

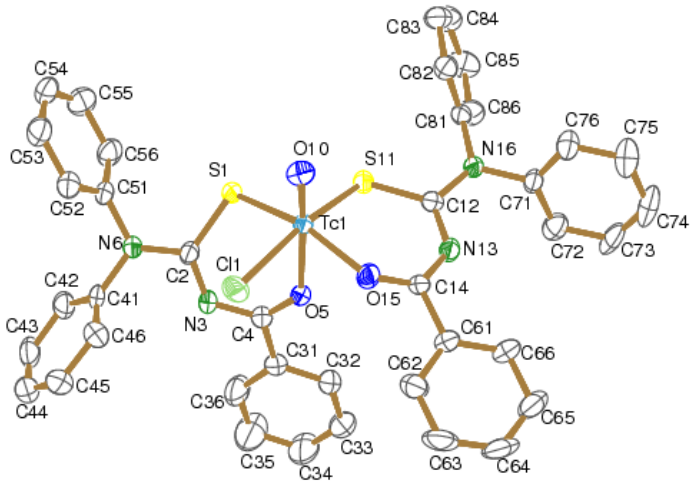

Figure 7 Ellipsoid plot (50% probability) of [TcOCl(Ph_2btu)$_2$].

8 [$ReCl_2(PPh_3)_2(Ph_2btu)$], (6a)

Table 8.1 Crystal data and structure refinement for [$ReCl_2(PPh_3)_2(Ph_2btu)$].

Empirical formula	$C_{56}H_{45}Cl_2N_2OP_2ReS$	
Formula weight	1113.04	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 12.689(1) Å	α= 77.87(1)°
	b = 13.334(1) Å	β= 85.38(1)°
	c = 15.004(1) Å	γ = 74.62(1)°
Volume	2392.3(3) $Å^3$	
Z	2	
Density (calculated)	1.545 g/cm^3	
Absorption coefficient	2.805 mm^{-1}	
F(000)	1116	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.350 x 0.213 x 0.030 mm^3	
Theta range for data collection	1.66 to 29.19	
Index ranges	-17<=h<=17, -18<=k<=18, -20<=l<=20	
Reflections collected	25265	
Independent reflections	12731 [R(int) = 0.0578]	
Completeness to theta = 29.19°	98.3 %	
Absorption correction	Integration	
Max. and min. transmission	0.5956 and 0.4629	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12731 / 0 / 586	
Goodness-of-fit on F^2	0.978	
Final R indices [I>2sigma(I)]	R1 = 0.0353, wR2 = 0.0851	
R indices (all data)	R1 = 0.0471, wR2 = 0.1004	
Largest diff. peak and hole	0.795 and -3.499 $e.Å^{-3}$	

Table 8.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[ReCl_2(PPh_3)_2(Ph_2btu)]$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	5835(1)	7536(1)	7011(1)	25(1)
Cl(1)	5211(1)	5976(1)	7149(1)	36(1)
Cl(2)	5018(1)	8527(1)	5611(1)	40(1)
S(1)	6710(1)	6758(1)	8410(1)	31(1)
C(2)	7304(3)	7632(3)	8746(2)	30(1)
N(3)	7417(3)	8585(3)	8310(2)	32(1)
C(4)	7002(3)	9095(3)	7506(2)	27(1)
O(5)	6439(2)	8797(2)	6999(2)	29(1)
C(31)	7206(3)	10159(3)	7150(2)	27(1)
C(32)	6663(3)	10812(3)	6384(3)	36(1)
C(33)	6810(4)	11816(3)	6074(3)	41(1)
C(34)	7505(4)	12179(3)	6524(3)	42(1)
C(35)	8055(4)	11539(4)	7272(3)	41(1)
C(36)	7909(3)	10525(3)	7592(3)	33(1)
N(6)	7736(3)	7301(3)	9594(2)	35(1)
C(41)	8394(3)	7861(3)	9941(3)	35(1)
C(42)	8081(4)	8208(4)	10758(3)	46(1)
C(43)	8769(5)	8654(4)	11137(4)	58(1)
C(44)	9746(5)	8757(4)	10698(4)	62(2)
C(45)	10029(5)	8434(5)	9906(4)	61(1)
C(46)	9358(4)	7985(4)	9514(3)	48(1)
C(51)	7627(4)	6321(4)	10169(3)	42(1)
C(52)	8405(5)	5410(5)	10113(5)	74(2)
C(53)	8286(8)	4460(6)	10707(8)	116(4)
C(54)	7419(9)	4506(8)	11334(6)	112(4)
C(55)	6665(9)	5401(8)	11353(4)	96(3)
C(56)	6745(6)	6325(5)	10774(3)	63(2)
P(1)	7422(1)	6671(1)	6114(1)	28(1)
C(61)	8474(3)	5711(3)	6837(3)	37(1)
C(62)	8234(5)	4799(4)	7340(4)	56(1)
C(63)	8988(6)	4079(5)	7921(5)	75(2)
C(64)	9981(7)	4238(6)	8000(6)	92(3)
C(65)	10184(8)	5139(8)	7565(8)	131(5)
C(66)	9457(6)	5887(6)	6967(6)	95(3)

C(71)	8207(3)	7496(3)	5353(3)	39(1)
C(72)	9054(6)	7046(6)	4807(7)	115(4)
C(73)	9679(7)	7664(6)	4287(8)	134(5)
C(74)	9459(5)	8730(5)	4267(5)	74(2)
C(75)	8570(6)	9188(5)	4738(4)	64(2)
C(76)	7938(5)	8575(4)	5279(3)	52(1)
C(81)	7142(3)	5866(3)	5347(3)	36(1)
C(82)	6149(4)	6114(4)	4940(3)	40(1)
C(83)	5984(4)	5537(4)	4310(3)	46(1)
C(84)	6795(5)	4716(5)	4096(4)	65(2)
C(85)	7786(5)	4421(6)	4535(5)	73(2)
C(86)	7953(4)	4985(5)	5164(4)	59(1)
P(2)	4167(1)	8383(1)	7810(1)	30(1)
C(91)	3972(3)	7872(4)	9026(3)	36(1)
C(92)	3483(4)	8520(5)	9638(3)	49(1)
C(93)	3266(5)	8084(6)	10541(4)	58(1)
C(94)	3523(5)	7000(6)	10825(3)	64(2)
C(95)	3983(5)	6339(5)	10228(3)	55(1)
C(96)	4206(4)	6776(4)	9329(3)	41(1)
C(101)	4105(3)	9784(3)	7726(3)	36(1)
C(102)	4699(4)	10097(4)	8312(4)	44(1)
C(103)	4729(4)	11159(4)	8174(4)	53(1)
C(104)	4172(5)	11892(4)	7475(4)	60(1)
C(105)	3593(5)	11591(4)	6887(4)	61(1)
C(106)	3563(4)	10534(4)	7006(3)	47(1)
C(111)	2838(3)	8405(3)	7384(3)	33(1)
C(112)	1894(4)	8884(4)	7816(3)	49(1)
C(113)	870(4)	8920(5)	7524(4)	59(1)
C(114)	785(4)	8467(4)	6796(4)	56(1)
C(115)	1718(5)	8004(5)	6362(5)	63(2)
C(116)	2743(4)	7964(4)	6651(4)	49(1)

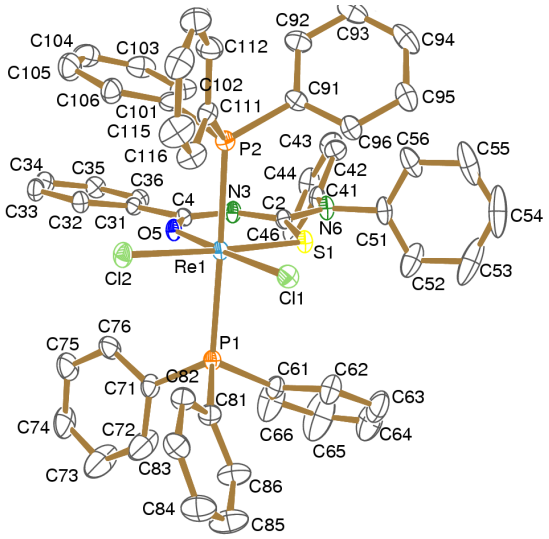

Figure 8 Ellipsoid plot (50% probability) of $[ReCl_2(PPh_3)_2(Ph_2btu)]$.

9 [TcCl(PPh$_3$)(Ph$_2$btu)$_2$], (7a)

Table 9.1 Crystal data and structure refinement for [TcCl(PPh$_3$)(Ph$_2$btu)$_2$]·MeOH.

Empirical formula	$C_{59}H_{49}ClN_4O_3PS_2Tc$	
Formula weight	1090.56	
Temperature	200(2) K	
Wavelength	0.71069 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 12.729(5) Å	α= 73.34(1)°
	b = 13.286(5) Å	β= 80.06(1)°
	c = 17.181(5) Å	γ = 89.27(1)°
Volume	2739.9(2) Å^3	
Z	2	
Density (calculated)	1.322 g/cm^3	
Absorption coefficient	0.464 mm^{-1}	
F(000)	1124	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.3 x 0.2 x 0.15 mm^3	
Theta range for data collection	1.87 to 25.00	
Index ranges	-15<=h<=15, -15<=k<=15, -20<=l<=20	
Reflections collected	34277	
Independent reflections	9407 [R(int) = 0.2136]	
Completeness to theta = 25.00°	97.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9407 / 0 / 642	
Goodness-of-fit on F^2	0.839	
Final R indices [I>2sigma(I)]	R1 = 0.0695, wR2 = 0.0947	
R indices (all data)	R1 = 0.1619, wR2 = 0.1185	
Largest diff. peak and hole	0.441 and -0.477 e.Å^{-3}	

Table 9.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[TcCl(PPh_3)(Ph_2btu)_2]\cdot MeOH$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	5714(1)	6379(1)	7441(1)	26(1)
Cl	7496(1)	5873(2)	7656(1)	46(1)
S(1)	4008(1)	6851(2)	7183(1)	37(1)
C(2)	3923(5)	6958(5)	6171(5)	33(2)
N(3)	4642(4)	6811(4)	5563(4)	31(2)
C(4)	5642(5)	6503(5)	5629(4)	28(2)
O(5)	6108(3)	6376(4)	6237(3)	34(1)
N(6)	2934(4)	7242(4)	5969(4)	31(1)
C(31)	6290(5)	6388(5)	4855(5)	36(2)
C(32)	5778(5)	6209(6)	4224(4)	40(2)
C(33)	6395(7)	6064(7)	3533(5)	56(2)
C(34)	7485(8)	6104(7)	3424(5)	58(2)
C(35)	7976(6)	6294(6)	4030(6)	49(2)
C(36)	7399(6)	6408(6)	4749(5)	45(2)
C(41)	2637(5)	7235(7)	5194(5)	42(2)
C(42)	2099(5)	8067(6)	4788(5)	44(2)
C(43)	1720(6)	8023(8)	4084(6)	63(3)
C(44)	1914(7)	7223(9)	3760(6)	70(3)
C(45)	2466(6)	6362(7)	4171(5)	55(2)
C(46)	2810(5)	6373(6)	4896(5)	41(2)
C(51)	2102(6)	7536(6)	6544(5)	37(2)
C(52)	2174(6)	8518(6)	6671(5)	43(2)
C(53)	1313(7)	8790(8)	7195(5)	60(3)
C(54)	438(6)	8102(9)	7548(5)	56(3)
C(55)	388(6)	7178(8)	7396(5)	54(2)
C(56)	1232(5)	6847(6)	6911(5)	41(2)
S(11)	5050(2)	6322(2)	8823(1)	37(1)
C(12)	4038(5)	5382(6)	9033(4)	36(2)
N(13)	4146(5)	4435(5)	8913(4)	37(2)
C(14)	4816(5)	4173(5)	8333(5)	31(2)
O(15)	5330(4)	4799(4)	7668(4)	42(1)
N(16)	3056(4)	5593(5)	9359(4)	34(2)
C(61)	4959(5)	3033(6)	8449(5)	37(2)
C(62)	5589(6)	2694(6)	7852(6)	49(2)

C(63)	5729(7)	1630(7)	7962(6)	62(3)
C(64)	5239(8)	899(7)	8671(6)	61(3)
C(65)	4602(8)	1232(7)	9272(7)	78(3)
C(66)	4454(6)	2302(6)	9169(6)	55(2)
C(71)	2124(6)	4915(6)	9439(5)	36(2)
C(72)	1834(6)	4804(7)	8729(6)	56(2)
C(73)	919(6)	4202(8)	8802(6)	64(3)
C(74)	327(7)	3725(7)	9543(7)	59(2)
C(75)	623(6)	3850(7)	10234(7)	59(3)
C(76)	1551(6)	4453(7)	10187(6)	54(2)
C(81)	2831(5)	6549(6)	9568(5)	38(2)
C(82)	2245(5)	7307(6)	9098(5)	45(2)
C(83)	2000(6)	8215(6)	9307(6)	57(2)
C(84)	2337(8)	8411(8)	9983(8)	89(4)
C(85)	2898(10)	7629(11)	10453(8)	123(6)
C(86)	3143(8)	6709(9)	10251(7)	89(4)
P	6387(1)	8184(2)	7100(1)	34(1)
C(91)	7087(6)	8569(6)	7820(5)	41(2)
C(92)	7464(7)	9594(6)	7636(6)	59(3)
C(93)	7986(8)	9941(8)	8129(7)	73(3)
C(94)	8139(8)	9301(9)	8871(7)	73(3)
C(95)	7766(6)	8230(8)	9098(6)	60(3)
C(96)	7242(6)	7873(6)	8574(5)	46(2)
C(101)	7365(6)	8528(6)	6168(5)	36(2)
C(102)	7057(6)	8709(6)	5393(5)	42(2)
C(103)	7768(7)	8919(6)	4655(6)	56(2)
C(104)	8853(7)	8938(8)	4682(6)	72(3)
C(105)	9200(6)	8740(7)	5440(6)	67(3)
C(106)	8461(6)	8537(6)	6171(6)	53(2)
C(111)	5431(5)	9234(6)	6955(5)	39(2)
C(112)	4520(6)	9112(6)	7567(5)	47(2)
C(113)	3791(5)	9906(6)	7512(6)	50(2)
C(114)	3992(7)	10834(6)	6853(6)	53(2)
C(115)	4845(6)	10943(6)	6268(5)	46(2)
C(116)	5587(6)	10150(6)	6298(5)	45(2)
C(120)	-251(10)	5664(12)	5825(9)	122(5)
O(121)	-489(7)	5006(8)	6723(8)	138(4)

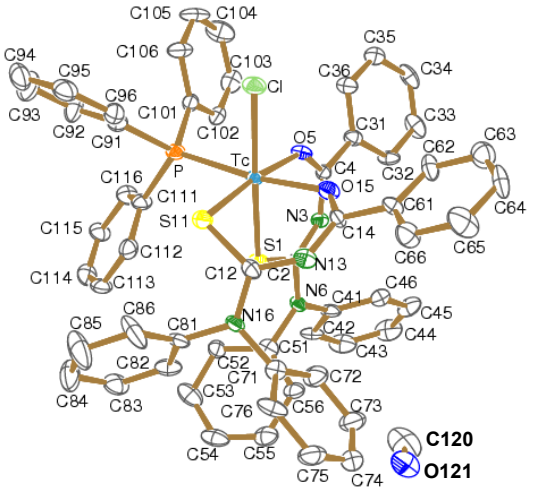

Figure 9 Ellipsoid plot (50% probability) of $[TcCl(PPh_3)(Ph_2btu)_2]\cdot MeOH$.

10 [$Tc(Ph_2btu)_3$], (8a)

Table 10.1 Crystal data and structure refinement for $[Tc(Ph_2btu)_3]\cdot MeOH$.

Empirical formula	$C_{61}H_{49}N_6O_4S_3Tc$	
Formula weight	1124.24	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 12.999(1) Å	α= 93.08(1)°
	b = 13.635(1) Å	β= 106.80(1)°
	c = 16.615(2) Å	γ = 95.57(1)°
Volume	2795.6(4) $Å^3$	
Z	2	
Density (calculated)	1.336 g/cm^3	
Absorption coefficient	0.422 mm^{-1}	
F(000)	1160	
Crystal description	Block	
Crystal color	Dark red	
Crystal size	0.450 x 0.317 x 0.250 mm^3	
Theta range for data collection	1.51 to 26.86	
Index ranges	-16<=h<=15, -17<=k<=17, -21<=l<=17	
Reflections collected	24178	
Independent reflections	11749 [R(int) = 0.0915]	
Completeness to theta = 26.86°	97.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11749 / 0 / 698	
Goodness-of-fit on F^2	0.888	
Final R indices [I>2sigma(I)]	R1 = 0.0588, wR2 = 0.1335	
R indices (all data)	R1 = 0.1291, wR2 = 0.1697	
Largest diff. peak and hole	0.935 and -0.911 $e.Å^{-3}$	

Table 10.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[Tc(Ph_2btu)_3]$·MeOH. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	6472(1)	7696(1)	7438(1)	37(1)
S(1)	7824(1)	6665(1)	7494(1)	45(1)
C(2)	8789(5)	7247(4)	7088(4)	40(1)
N(3)	8841(4)	8120(3)	6774(3)	39(1)
C(4)	8162(4)	8798(4)	6749(3)	37(1)
O(5)	7340(3)	8779(2)	7013(2)	41(1)
C(31)	8413(5)	9706(4)	6343(3)	39(1)
C(32)	7706(5)	10425(4)	6215(4)	49(2)
C(33)	7935(6)	11271(4)	5835(5)	61(2)
C(34)	8872(6)	11422(4)	5604(5)	59(2)
C(35)	9586(6)	10710(4)	5746(4)	54(2)
C(36)	9368(5)	9855(4)	6120(4)	48(2)
N(6)	9643(4)	6726(3)	7103(3)	42(1)
C(41)	10612(5)	7147(4)	6935(4)	45(1)
C(42)	11005(5)	6620(4)	6375(4)	51(2)
C(43)	11985(5)	6966(5)	6260(5)	60(2)
C(44)	12545(6)	7820(5)	6692(6)	70(2)
C(45)	12147(6)	8367(5)	7241(5)	71(2)
C(46)	11188(5)	8022(5)	7375(5)	57(2)
C(51)	9607(5)	5677(4)	7242(4)	48(2)
C(52)	8985(5)	4991(4)	6596(5)	57(2)
C(53)	9009(7)	3997(5)	6700(7)	77(3)
C(54)	9655(8)	3715(6)	7456(8)	86(3)
C(55)	10272(7)	4380(6)	8080(6)	84(3)
C(56)	10243(6)	5405(5)	7981(5)	64(2)
S(11)	5410(1)	6417(1)	7826(1)	42(1)
C(12)	4445(4)	5846(4)	6924(4)	36(1)
N(13)	4252(4)	6042(3)	6126(3)	37(1)
C(14)	4830(5)	6694(4)	5817(4)	38(1)
O(15)	5678(3)	7278(3)	6194(2)	42(1)
C(61)	4443(4)	6762(4)	4895(4)	38(1)
C(62)	3844(5)	5951(4)	4352(4)	47(2)
C(63)	3549(5)	6000(5)	3494(4)	53(2)
C(64)	3821(5)	6857(5)	3160(4)	56(2)

C(65)	4401(5)	7670(5)	3685(4)	54(2)
C(66)	4728(5)	7614(4)	4542(4)	42(1)
N(16)	3786(4)	5068(3)	7065(3)	39(1)
C(71)	3020(5)	4437(4)	6376(3)	37(1)
C(72)	3377(5)	3941(4)	5766(4)	42(1)
C(73)	2658(5)	3309(4)	5153(4)	44(1)
C(74)	1591(5)	3129(4)	5130(4)	45(1)
C(75)	1240(5)	3614(4)	5739(4)	50(2)
C(76)	1967(5)	4250(4)	6372(4)	44(1)
C(81)	3834(5)	4791(4)	7888(4)	42(1)
C(82)	3222(6)	5215(5)	8336(4)	61(2)
C(83)	3223(7)	4873(6)	9108(5)	76(2)
C(84)	3810(8)	4132(6)	9416(5)	84(3)
C(85)	4415(8)	3710(6)	8961(5)	84(3)
C(86)	4432(6)	4042(5)	8208(4)	63(2)
S(21)	7277(1)	8281(1)	8855(1)	46(1)
C(22)	6813(5)	9403(4)	9030(4)	45(1)
N(23)	6069(4)	9868(3)	8507(3)	44(1)
C(24)	5411(5)	9524(4)	7750(3)	38(1)
O(25)	5373(3)	8696(3)	7338(2)	41(1)
C(91)	4591(5)	10172(4)	7341(4)	42(1)
C(92)	4034(5)	9990(4)	6500(4)	51(2)
C(93)	3232(6)	10576(5)	6120(5)	61(2)
C(94)	3005(6)	11332(5)	6610(5)	65(2)
C(95)	3566(6)	11522(4)	7445(5)	57(2)
C(96)	4355(6)	10967(4)	7816(4)	52(2)
N(26)	7249(5)	9863(3)	9816(3)	52(1)
C(101)	6857(6)	10770(4)	10059(4)	53(2)
C(102)	6177(8)	10740(5)	10535(6)	84(3)
C(103)	5841(8)	11625(6)	10792(5)	83(3)
C(104)	6200(8)	12463(5)	10568(5)	83(2)
C(105)	6850(10)	12508(6)	10057(9)	130(5)
C(106)	7179(9)	11631(5)	9794(7)	113(4)
C(111)	8084(6)	9510(4)	10470(4)	54(2)
C(112)	7852(7)	8765(6)	10919(5)	78(2)
C(113)	8674(9)	8482(7)	11587(6)	91(3)
C(114)	9687(9)	8947(6)	11783(5)	88(3)
C(115)	9904(8)	9693(6)	11346(6)	100(3)
C(116)	9105(8)	9991(5)	10682(6)	88(3)

C(121)	2290(9)	5368(9)	1111(7)	43(3)
O(122)	1575(6)	5260(5)	348(5)	40(2)
O(124)	-48(8)	6360(7)	-16(7)	68(3)
C(123)	-134(12)	7239(8)	-144(9)	62(4)

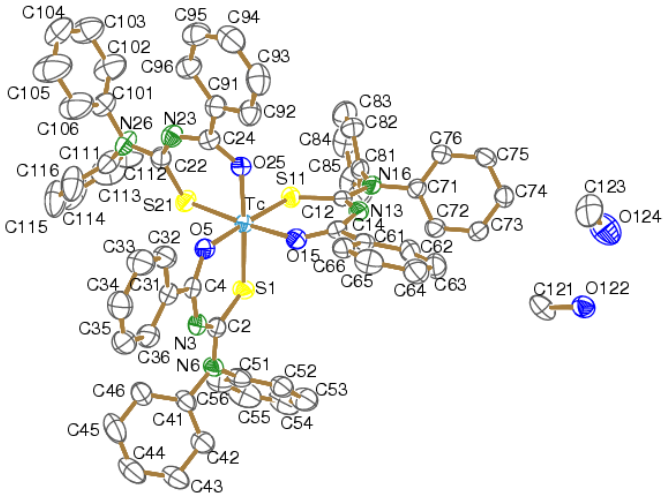

Figure 10 Ellipsoid plot (50% probability) of $[Tc(Ph_2btu)_3]\cdot MeOH$.

11 [Re(NPh)Br_2(Ph_2btu)(PPh_3)], (9a)

Table 11.1 Crystal data and structure refinement for [Re(NPh)Br_2(Ph_2btu)(PPh_3)].

Empirical formula	$C_{44}H_{35}Br_2N_3OPReS$	
Formula weight	1030.80	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 12.208(1) Å	α= 90°
	b = 19.265(1) Å	β= 93.59(1)°
	c = 17.257(1) Å	γ = 90°
Volume	4050.8(5) $Å^3$	
Z	4	
Density (calculated)	1.690 g/cm^3	
Absorption coefficient	5.101 mm^{-1}	
F(000)	2016	
Crystal description	Plate	
Crystal color	Orange	
Crystal size	0.370 x 0.177 x 0.040 mm^3	
Theta range for data collection	2.70 to 26.86	
Index ranges	-15<=h<=15, -24<=k<=22, -16<=l<=21	
Reflections collected	19089	
Independent reflections	8559 [R(int) = 0.0577]	
Completeness to theta = 26.86°	98.2 %	
Absorption correction	Integration	
Max. and min. transmission	0.5224 and 0.2917	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8559 / 0 / 478	
Goodness-of-fit on F^2	1.020	
Final R indices [I>2sigma(I)]	R1 = 0.0522, wR2 = 0.1281	
R indices (all data)	R1 = 0.0684, wR2 = 0.1361	
Largest diff. peak and hole	2.289 and -3.837 $e.Å^{-3}$	

Table 11.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Re(NPh)Br_2(Ph_2btu)(PPh_3)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	6729(1)	9809(1)	1986(1)	24(1)
Br(1)	5979(1)	8849(1)	1099(1)	34(1)
Br(2)	8686(1)	9518(1)	1561(1)	39(1)
S(1)	7508(1)	10479(1)	3068(1)	30(1)
C(2)	8625(5)	9959(4)	3375(4)	27(1)
N(3)	8558(4)	9302(3)	3575(4)	32(1)
C(4)	7765(5)	8881(3)	3310(4)	27(1)
O(5)	7050(3)	8998(2)	2749(3)	26(1)
C(31)	7713(5)	8194(4)	3685(4)	31(1)
C(32)	7071(5)	7670(4)	3342(5)	34(2)
C(33)	7004(6)	7019(4)	3687(6)	46(2)
C(34)	7561(8)	6904(5)	4395(6)	56(2)
C(35)	8207(10)	7409(5)	4740(6)	71(3)
C(36)	8304(8)	8056(4)	4380(5)	53(2)
N(6)	9618(4)	10275(3)	3510(4)	30(1)
C(41)	10550(5)	9876(4)	3837(4)	30(1)
C(42)	10841(5)	9259(4)	3480(5)	38(2)
C(43)	11734(6)	8887(5)	3799(7)	56(2)
C(44)	12337(6)	9151(6)	4440(6)	53(3)
C(45)	12064(6)	9756(5)	4749(5)	46(2)
C(46)	11163(6)	10124(5)	4452(5)	39(2)
C(51)	9800(5)	10987(4)	3319(5)	34(2)
C(52)	10343(6)	11131(5)	2665(5)	42(2)
C(53)	10551(7)	11829(6)	2488(7)	60(3)
C(54)	10247(7)	12340(5)	2966(7)	62(3)
C(55)	9694(8)	12196(5)	3610(7)	62(3)
C(56)	9478(7)	11512(5)	3802(6)	50(2)
N(10)	6678(4)	10525(3)	1385(3)	27(1)
C(61)	6958(5)	11105(4)	986(4)	29(1)
C(62)	7933(7)	11448(5)	1195(6)	50(2)
C(63)	8264(8)	11992(5)	746(7)	62(3)
C(64)	7598(8)	12224(5)	128(6)	58(2)
C(65)	6620(7)	11893(4)	-74(5)	45(2)
C(66)	6286(6)	11343(4)	347(4)	36(2)

P	4907(1)	9915(1)	2455(1)	24(1)
C(71)	4807(5)	9282(4)	3238(4)	30(1)
C(72)	5219(5)	9466(4)	3976(4)	36(2)
C(73)	5298(6)	8966(5)	4562(4)	39(2)
C(74)	4985(6)	8299(5)	4410(5)	43(2)
C(75)	4567(6)	8110(4)	3673(5)	42(2)
C(76)	4502(5)	8601(4)	3083(5)	34(2)
C(81)	3729(5)	9783(3)	1770(4)	27(1)
C(82)	2763(5)	9481(5)	1988(5)	39(2)
C(83)	1848(6)	9470(5)	1456(5)	48(2)
C(84)	1886(6)	9762(5)	737(5)	45(2)
C(85)	2857(6)	10059(4)	502(5)	38(2)
C(86)	3774(5)	10068(4)	1024(4)	30(1)
C(91)	4525(5)	10734(4)	2904(4)	30(1)
C(92)	5149(6)	11338(4)	2853(4)	34(2)
C(93)	4781(7)	11952(4)	3187(5)	42(2)
C(94)	3825(7)	11967(5)	3553(5)	44(2)
C(95)	3191(6)	11372(4)	3601(5)	42(2)
C(96)	3545(5)	10758(4)	3281(5)	36(2)

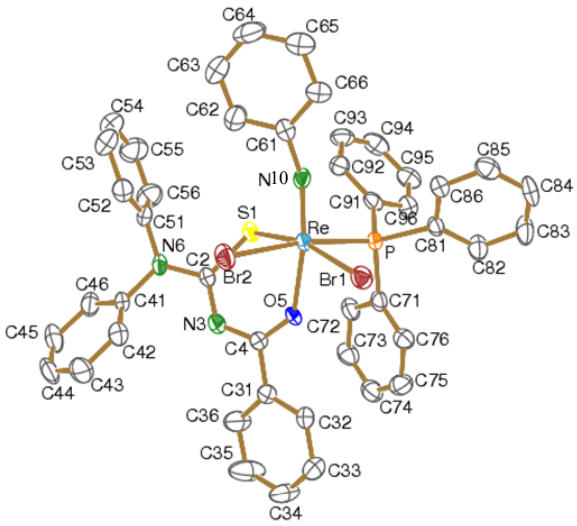

Figure 11 Ellipsoid plot (50% probability) of [Re(NPh)Br_2(Ph_2btu)(PPh_3)].

12 $(NBu_4)_2[\{Re_2O_2Cl_5(Hpicbtu)\}_2O]$, (10)

Table 12.1 Crystal data and structure refinement for $(NBu_4)_2[\{Re_2O_2Cl_5(Hpicbtu)\}_2O]\cdot Me_2CO$.

Empirical formula	$C_{63}H_{102}Cl_{10}N_8O_8Re_4S_2$	
Formula weight	2262.95	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 11.446(1) Å	α= 94.17(1)°
	b = 12.708(1) Å	β= 104.08(1)°
	c = 15.673(2) Å	γ = 97.79(1)°
Volume	2177.4(4) $Å^3$	
Z	1	
Density (calculated)	1.726 g/cm^3	
Absorption coefficient	5.945 mm^{-1}	
F(000)	1102	
Crystal description	Block	
Crystal color	Blue	
Crystal size	0.15 x 0.137 x 0.13 mm^3	
Theta range for data collection	2.22 to 29.32	
Index ranges	-15<=h<=15, -17<=k<=12, -21<=l<=21	
Reflections collected	20896	
Independent reflections	11339 [R(int) = 0.0701]	
Completeness to theta = 29.32°	94.9 %	
Absorption correction	Integration	
Max. and min. transmission	0.6017 and 0.3416	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11339 / 0 / 449	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0504, wR2 = 0.1220	
R indices (all data)	R1 = 0.0832, wR2 = 0.1379	
Extinction coefficient	0.0069(4)	
Largest diff. peak and hole	1.815 and -2.381 e.$Å^{-3}$	

Table 12.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $(NBu_4)_2[\{Re_2O_2Cl_5(Hpicbtu)\}_2O]\cdot Me_2CO$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	7426(1)	9231(1)	8253(1)	43(1)
O(10)	8406(6)	8911(5)	9111(4)	60(2)
Cl(1)	8781(2)	10483(2)	7769(2)	58(1)
Cl(2)	6716(2)	10687(2)	8859(2)	64(1)
Cl(3)	5749(2)	8068(2)	8491(2)	59(1)
S(1)	7654(2)	8018(1)	7121(1)	41(1)
C(2)	6197(6)	7256(6)	6689(5)	39(2)
N(3)	5247(5)	7810(5)	6367(4)	39(1)
C(4)	5223(6)	8864(6)	6506(5)	38(2)
O(5)	5966(4)	9503(4)	7099(3)	41(1)
C(31)	4241(6)	9292(6)	5906(5)	38(2)
C(32)	3764(6)	10142(6)	6232(6)	45(2)
C(33)	2914(8)	10598(7)	5675(7)	58(2)
C(34)	2513(8)	10213(8)	4774(7)	60(2)
C(35)	2995(7)	9367(8)	4446(6)	56(2)
C(36)	3834(6)	8910(7)	5016(5)	46(2)
Re(2)	4335(1)	5103(1)	6004(1)	42(1)
O(20)	3879(5)	5303(5)	6948(4)	62(2)
O(30)	5000	5000	5000	39(2)
Cl(4)	2784(2)	3640(2)	5298(2)	56(1)
Cl(5)	3030(2)	6244(2)	5208(2)	56(1)
N(6)	5992(5)	6237(4)	6559(4)	40(1)
C(7)	7060(7)	5689(6)	6818(6)	51(2)
C(41)	6717(7)	4545(6)	6940(5)	45(2)
C(42)	7572(8)	3992(7)	7399(6)	52(2)
C(43)	7252(9)	2927(7)	7483(6)	61(2)
C(44)	6074(9)	2452(7)	7114(6)	61(2)
C(45)	5237(9)	3047(7)	6675(6)	57(2)
N(46)	5560(6)	4095(5)	6601(4)	46(2)
N(50)	8209(6)	12098(5)	11469(4)	44(1)
C(51)	8213(7)	10928(7)	11207(6)	49(2)
C(52)	7212(9)	10147(7)	11395(7)	62(2)
C(53)	7360(11)	8997(8)	11132(7)	72(3)

C(54)	6444(12)	8198(9)	11355(10)	93(4)
C(61)	9392(7)	12734(7)	11369(5)	48(2)
C(62)	9530(8)	12794(8)	10447(6)	57(2)
C(63)	10731(9)	13450(8)	10454(6)	59(2)
C(64)	10959(12)	13472(10)	9538(8)	87(4)
C(71)	7118(7)	12459(7)	10880(6)	51(2)
C(72)	7021(8)	13629(7)	11043(6)	56(2)
C(73)	6008(9)	13931(8)	10330(6)	60(2)
C(74)	6268(10)	13941(9)	9413(7)	73(3)
C(81)	8162(7)	12290(7)	12433(5)	46(2)
C(82)	9146(7)	11903(8)	13120(6)	56(2)
C(83)	8947(7)	12070(7)	14027(6)	52(2)
C(84)	9872(8)	11621(9)	14710(6)	64(2)
C(91)	10273(13)	4580(13)	6061(9)	42(3)
C(92)	10650(10)	4329(10)	7000(8)	27(2)
C(93)	11290(20)	3442(16)	7181(11)	64(5)
O(94)	10463(8)	4876(7)	7596(6)	36(2)

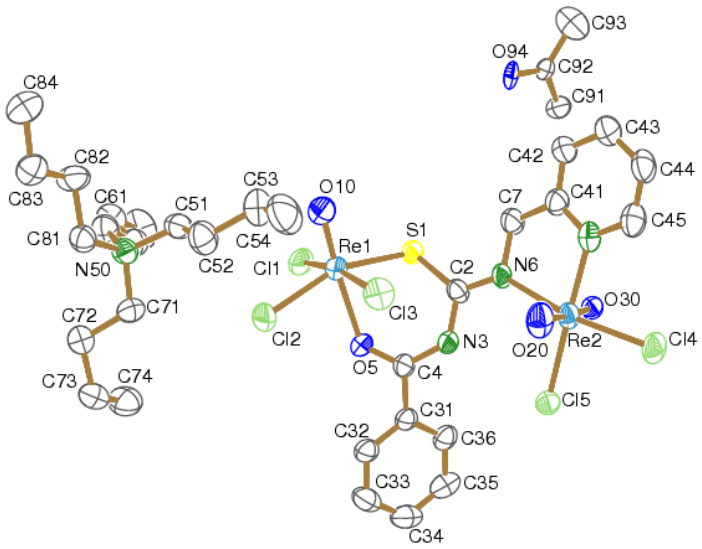

Figure 12 Ellipsoid plot (50% probability) of the asymmetric unit of $(NBu_4)_2[\{Re_2O_2Cl_5(Hpicbtu)\}_2O]\cdot Me_2CO$.

13 [{ReO(OMe)Phth(Et_2tu)$_2$}$_2$], (11a)

Table 13.1 Crystal data and structure refinement for [{ReO(OMe)Phth(Et_2tu)$_2$}$_2$]·3/2CH_2Cl_2.

Empirical formula	$C_{20.50}H_{27}Cl_3N_4O_4ReS_2$	
Formula weight	750.13	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 27.336(1) Å	α= 90°
	b = 8.246(1) Å	β= 96.57(1)°
	c = 25.021(1) Å	γ = 90°
Volume	5602.7(7) Å^3	
Z	8	
Density (calculated)	1.779 g/cm^3	
Absorption coefficient	4.806 mm^{-1}	
F(000)	2944	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.100 mm^3	
Theta range for data collection	1.50 to 26.88	
Index ranges	-34<=h<=34, -10<=k<=9, -27<=l<=31	
Reflections collected	14446	
Independent reflections	5904 [R(int) = 0.0629]	
Completeness to theta = 26.88°	97.6 %	
Absorption correction	Integration	
Max. and min. transmission	0.6276 and 0.2339	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5904 / 0 / 313	
Goodness-of-fit on F^2	0.929	
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1401	
R indices (all data)	R1 = 0.0590, wR2 = 0.1582	
Extinction coefficient	0.00091(10)	
Largest diff. peak and hole	5.438 and -2.419 e.Å^{-3}	

Table 13.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(OMe)Phth(Et_2tu)$_2$}$_2$]·3/2CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	1326(1)	2796(1)	8321(1)	31(1)
O(10)	1265(2)	4861(7)	8297(2)	40(1)
O(20)	1211(2)	564(6)	8218(2)	35(1)
C(20)	819(3)	-420(11)	7953(3)	51(2)
S(1)	2131(1)	2637(2)	8113(1)	34(1)
C(2)	2114(3)	2400(8)	7420(3)	31(1)
N(3)	1723(2)	2006(6)	7057(2)	29(1)
C(4)	1253(2)	2252(7)	7107(3)	28(1)
O(5)	1041(2)	2846(6)	7490(2)	37(1)
C(31)	920(2)	1848(7)	6609(2)	27(1)
C(32)	1111(2)	1260(9)	6153(2)	36(1)
C(33)	800(2)	955(10)	5692(3)	42(2)
C(36)	412(2)	2130(7)	6582(3)	30(1)
N(6)	2542(2)	2486(7)	7215(2)	32(1)
C(41)	2579(3)	2055(8)	6650(3)	36(1)
C(42)	2457(3)	3459(11)	6269(3)	49(2)
C(43)	2999(2)	3064(8)	7534(3)	33(1)
C(44)	3306(2)	1658(10)	7801(3)	43(2)
S(11)	1626(1)	2483(3)	9221(1)	41(1)
C(12)	1121(3)	2696(10)	9597(3)	40(2)
N(13)	647(2)	2382(8)	9422(3)	39(1)
C(14)	427(2)	2347(8)	8926(3)	32(1)
O(15)	580(2)	2743(6)	8482(2)	39(1)
C(131)	-109(2)	1896(8)	8888(2)	29(1)
C(132)	-307(2)	1251(9)	9337(2)	36(1)
N(16)	1226(2)	3048(8)	10112(2)	43(1)
C(141)	1725(3)	3372(13)	10365(3)	55(2)
C(142)	1967(4)	1862(16)	10592(4)	77(3)
C(143)	829(3)	3114(12)	10474(3)	55(2)
C(144)	638(5)	4709(16)	10507(5)	95(4)
C(51)	1287(5)	2241(14)	4190(4)	76(3)
Cl(1)	1678(1)	2491(5)	4791(2)	93(1)
Cl(2)	661(2)	2239(6)	4287(2)	111(2)
C(52)	0	-4537(18)	7500	75(4)
Cl(3)	205(1)	-3356(5)	6996(1)	88(1)

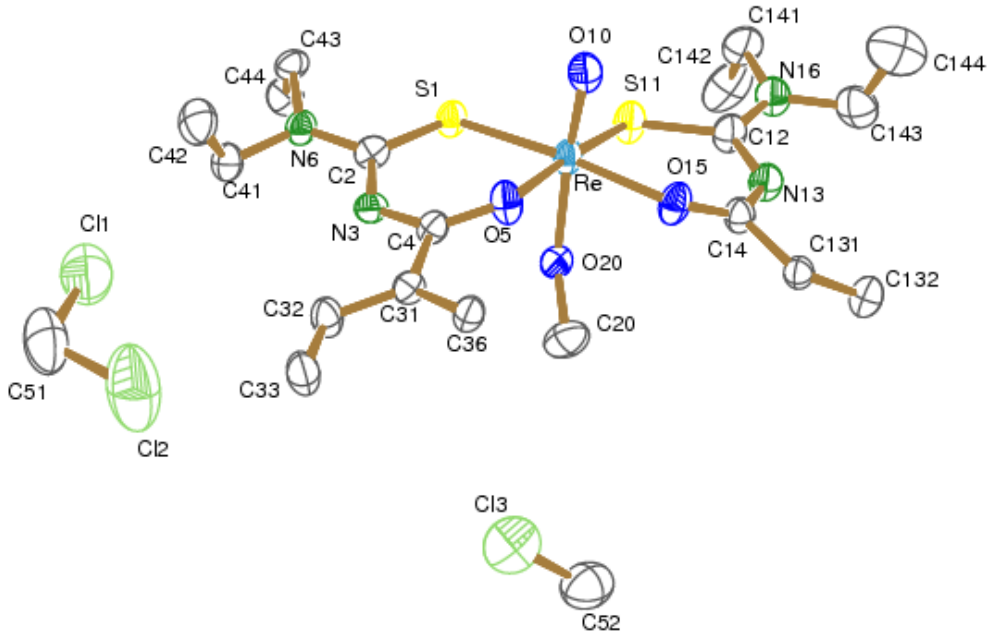

Figure 13 Ellipsoid plot (50% probability) of the asymmetric unit of $(NBu_4)_2[\{Re_2O_2Cl_5(Hpicbtu)\}_2O]\cdot Me_2CO$. C52 is placed at a special position which located on the glide (1-x,y,-z+3/2).

14 [{ReO(OMe)Phth(i-Bu_2tu)$_2$}$_2$], (11b)

Table 14.1 Crystal data and structure refinement for [{ReO(OMe)Phth(i-Bu_2tu)$_2$}$_2$].

Empirical formula	$C_{54}H_{80}N_8O_8Re_2S_4$	
Formula weight	1469.90	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.219(1) Å	α= 76.89(1)°
	b = 10.346(1) Å	β= 83.20(1)°
	c = 18.489(2) Å	γ = 64.15(1)°
Volume	1545.3(3) Å^3	
Z	1	
Density (calculated)	1.580 g/cm^3	
Absorption coefficient	4.104 mm^{-1}	
F(000)	738	
Crystal description	Plate	
Crystal color	Orange brown	
Crystal size	0.210 x 0.137 x 0.030 mm^3	
Theta range for data collection	2.23 to 29.27	
Index ranges	-12<=h<=12, -14<=k<=14, -25<=l<=23	
Reflections collected	16221	
Independent reflections	8237 [R(int) = 0.0736]	
Completeness to theta = 29.27°	97.5 %	
Absorption correction	Integration	
Max. and min. transmission	0.8209 and 0.4816	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8237 / 0 / 344	
Goodness-of-fit on F^2	1.008	
Final R indices [I>2sigma(I)]	R1 = 0.0578, wR2 = 0.1372	
R indices (all data)	R1 = 0.0858, wR2 = 0.1580	
Extinction coefficient	0.0177(13)	
Largest diff. peak and hole	4.839 and -1.679 e.Å^{-3}	

Table 14.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(OMe)Phth(i-Bu_2tu)$_2$}$_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	9373(1)	4431(1)	3029(1)	45(1)
O(10)	11245(7)	3055(6)	3199(4)	57(1)
O(20)	7331(7)	6023(6)	3037(3)	59(1)
C(20)	6160(20)	6939(19)	3400(9)	139(7)
S(1)	8310(3)	3063(3)	2601(1)	59(1)
C(2)	7328(10)	2472(8)	3381(4)	49(2)
N(3)	7841(7)	2082(6)	4078(3)	44(1)
C(4)	8473(8)	2706(7)	4409(4)	39(1)
O(5)	8772(7)	3809(6)	4149(3)	48(1)
C(31)	8706(7)	2110(7)	5222(4)	38(1)
C(32)	8411(9)	896(7)	5571(4)	44(1)
C(33)	9085(8)	2845(7)	5668(4)	39(1)
C(34)	8504(10)	430(8)	6322(4)	49(2)
N(6)	6004(8)	2320(7)	3286(4)	50(1)
C(41)	5122(10)	1820(9)	3934(5)	55(2)
C(42)	3796(11)	3052(11)	4270(6)	69(2)
C(43)	2933(13)	2339(16)	4871(8)	93(4)
C(44)	4410(12)	3938(12)	4580(7)	78(3)
C(45)	5239(11)	2701(10)	2571(5)	59(2)
C(46)	5487(17)	1411(12)	2239(7)	86(3)
C(47)	4690(20)	1975(15)	1478(8)	114(5)
C(48)	7090(20)	310(20)	2223(11)	135(7)
S(11)	9892(3)	5347(3)	1799(1)	63(1)
C(12)	10805(10)	6500(8)	1813(4)	48(2)
N(13)	10958(8)	7042(7)	2382(3)	46(1)
C(14)	10605(8)	6720(7)	3096(4)	42(1)
O(15)	10129(7)	5773(5)	3441(3)	50(1)
C(131)	10891(8)	7596(7)	3559(4)	41(1)
C(132)	11173(9)	8822(8)	3236(4)	47(2)
N(16)	11463(8)	6933(6)	1160(3)	44(1)
C(141)	11322(11)	6604(9)	473(5)	57(2)
C(142)	9989(12)	7883(10)	-37(5)	63(2)
C(143)	10020(18)	7467(14)	-779(7)	94(4)
C(144)	8359(15)	8358(17)	313(7)	103(4)

C(145)	12284(10)	7908(9)	1133(4)	53(2)
C(146)	13977(12)	7103(11)	1429(5)	63(2)
C(147)	14619(14)	8210(14)	1465(7)	84(3)
C(148)	15131(13)	6021(12)	959(7)	80(3)

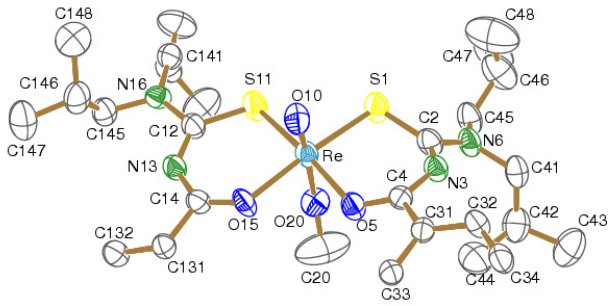

Figure 14 Ellipsoid plot (50% probability) of the asymmetric unit of [{ReO(OMe)Phth(i-Bu_2tu)$_2$}$_2$].

15 [({ReOphth(Et_2tu)$_2$}$_2$O)$_2$], (12a)

Table 15.1 Crystal data and structure refinement for [({ReOphth(Et_2tu)$_2$}$_2$O)$_2$].

Empirical formula	$C_{72}H_{96}N_{16}O_{14}Re_4S^8$	
Formula weight	2410.93	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 16.017(1) Å	α= 90°
	b = 18.621(1) Å	β= 117.83(1)°
	c = 16.055(1) Å	γ = 90°
Volume	4234.6(4) $Å^3$	
Z	2	
Density (calculated)	1.891 g/cm^3	
Absorption coefficient	5.966 mm^{-1}	
F(000)	2360	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.100 x 0.083 x 0.050 mm^3	
Theta range for data collection	1.80 to 29.28	
Index ranges	-21<=h<=21, -25<=k<=22, -21<=l<=21	
Reflections collected	26466	
Independent reflections	11357 [R(int) = 0.1465]	
Completeness to theta = 29.28°	98.4 %	
Absorption correction	Integration	
Max. and min. transmission	0.5149 and 0.3663	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11357 / 0 / 515	
Goodness-of-fit on F^2	0.912	
Final R indices [I>2sigma(I)]	R1 = 0.0650, wR2 = 0.1193	
R indices (all data)	R1 = 0.1625, wR2 = 0.1670	
Extinction coefficient	0.00290(12)	
Largest diff. peak and hole	1.751 and -1.706 e.$Å^{-3}$	

Table 15.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [({ReOphth(Et_2tu)$_2$}$_2$O)$_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	7796(1)	6036(1)	3363(1)	28(1)
O(10)	7926(8)	6548(7)	4283(8)	49(3)
O(20)	7912(6)	5371(6)	2523(6)	29(2)
S(1)	6176(3)	5853(2)	2671(3)	42(1)
C(2)	5992(9)	5080(9)	3164(10)	38(4)
N(3)	6546(8)	4800(7)	3976(9)	36(3)
C(4)	7478(10)	4864(8)	4512(10)	32(3)
O(5)	8080(7)	5125(6)	4280(7)	36(2)
C(41)	7852(9)	4553(8)	5448(10)	33(3)
C(42)	7290(12)	4125(11)	5682(12)	53(5)
N(6)	5119(9)	4783(8)	2677(9)	43(3)
C(51)	4869(12)	4177(12)	3131(13)	61(6)
C(52)	4625(16)	4493(17)	3887(15)	94(9)
C(53)	4422(11)	5032(11)	1758(11)	52(5)
C(54)	3753(13)	5597(13)	1771(15)	66(6)
S(11)	7444(2)	6969(2)	2295(3)	36(1)
C(12)	8523(11)	7188(8)	2337(11)	38(3)
N(13)	9376(8)	7150(7)	3073(9)	36(3)
C(14)	9689(9)	6691(8)	3802(9)	31(3)
O(15)	9292(6)	6143(5)	3919(6)	29(2)
C(234)	11215(10)	7368(9)	4370(11)	38(3)
C(235)	10665(9)	6869(8)	4554(10)	30(3)
C(236)	11032(9)	6536(8)	5414(10)	31(3)
N(16)	8453(8)	7489(7)	1542(9)	38(3)
C(151)	7579(10)	7594(9)	693(10)	40(4)
C(152)	7071(12)	8281(11)	674(13)	54(5)
C(153)	9356(13)	7736(9)	1526(12)	48(4)
C(154)	9586(13)	8460(10)	1923(14)	58(5)
Re(2)	8007(1)	4721(1)	1654(1)	27(1)
O(30)	8281(7)	4055(6)	1101(7)	39(2)
S(21)	6383(3)	4487(2)	937(3)	40(1)
C(22)	6285(10)	3676(10)	1446(11)	43(4)
N(23)	6812(9)	3458(8)	2341(9)	42(3)
C(24)	7704(9)	3621(7)	2932(10)	29(3)

O(25)	8279(6)	3991(6)	2762(6)	32(2)
C(43)	12397(14)	6190(13)	3429(13)	70(7)
C(44)	11486(12)	6047(12)	2744(12)	58(5)
C(45)	10885(11)	5627(9)	2958(10)	38(3)
C(46)	11221(9)	5349(7)	3850(9)	28(3)
N(26)	5575(9)	3245(9)	910(10)	52(4)
C(241)	5459(13)	2552(11)	1289(13)	57(5)
C(242)	4811(17)	2637(13)	1705(17)	79(7)
C(243)	4931(11)	3368(10)	-81(11)	47(4)
C(244)	5289(14)	2961(14)	-690(13)	74(6)
S(31)	7712(3)	5616(2)	527(3)	37(1)
C(32)	8814(11)	5895(7)	661(11)	35(3)
N(33)	9649(8)	5847(7)	1437(8)	35(3)
C(34)	9882(10)	5480(8)	2202(10)	30(3)
0(35)	9458(6)	4994(6)	2403(7)	33(2)
C(231)	8061(10)	3300(8)	3864(10)	31(3)
C(232)	7548(11)	2794(10)	4046(11)	47(4)
C(233)	7885(10)	2469(10)	4911(11)	52(5)
N(36)	8837(9)	6229(6)	-72(8)	35(3)
C(351)	8005(12)	6351(11)	-999(12)	54(5)
C(352)	8012(15)	5894(15)	-1737(13)	83(8)
C(353)	9744(10)	6489(9)	0(11)	38(3)
C(354)	10379(14)	5929(12)	-57(14)	63(6)

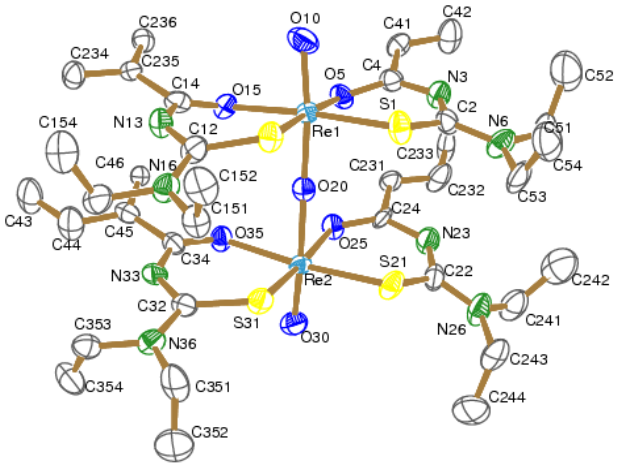

Figure 15 Ellipsoid plot (50% probability) of the asymmetric unit of $[(\{ReOphth(Et_2tu)_2\}_2O)_2]$.

16 [({ReOphth(*i*-Bu_2tu)$_2$}$_2$O)$_2$], (12b)

Table 16.1 Crystal data and structure refinement for [({ReOphth(*i*-Bu_2tu)$_2$}$_2$O)$_2$]·DMSO·H_2O.

Empirical formula	$C_{106}H_{164}N_{16}O_{16}Re_4S_9$	
Formula weight	3048.02	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 19.765(2) Å	α= 90°
	b = 15.811(1) Å	β= 92.86(1)°
	c = 23.642(2) Å	γ = 90°
Volume	7379.1(11) $Å^3$	
Z	2	
Density (calculated)	1.372 g/cm^3	
Absorption coefficient	3.468 mm^{-1}	
F(000)	3072	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.170 x 0.100 x 0.010 mm^3	
Theta range for data collection	1.65 to 29.34	
Index ranges	-27<=h<=27, -21<=k<=18, -21<=l<=32	
Reflections collected	51372	
Independent reflections	19660 [R(int) = 0.2109]	
Completeness to theta = 29.34°	97.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.7677 and 0.5523	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	19660 / 0 / 724	
Goodness-of-fit on F^2	0.948	
Final R indices [I>2sigma(I)]	R1 = 0.1033, wR2 = 0.2286	
R indices (all data)	R1 = 0.2173, wR2 = 0.2907	
Extinction coefficient	0.0067(4)	
Largest diff. peak and hole	4.580 and -1.927 e.$Å^{-3}$	

Table 16.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [({ReOphth(*i*-Bu_2tu)$_2$}$_2$O)$_2$]·DMSO·H_2O. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	9382(1)	9406(1)	1432(1)	41(1)
O(10)	10171(5)	9090(7)	1628(5)	52(3)
O(20)	8602(5)	9810(7)	1011(4)	48(2)
S(1)	8792(2)	8284(3)	1810(2)	47(1)
C(2)	8747(4)	7362(6)	1364(4)	15(2)
N(3)	9143(6)	7369(9)	879(5)	45(3)
C(4)	9512(7)	7948(11)	602(7)	46(3)
O(5)	9531(5)	8719(6)	655(4)	43(2)
C(41)	9970(7)	7574(10)	185(6)	43(3)
C(42)	10499(7)	8013(11)	7(6)	47(4)
C(43)	10952(8)	7639(10)	-359(6)	44(3)
C(44)	10844(8)	6807(11)	-538(7)	51(4)
C(45)	10275(9)	6382(12)	-385(7)	55(4)
C(46)	9856(8)	6742(10)	-10(6)	45(3)
N(6)	8345(7)	6823(9)	1407(6)	55(3)
C(51)	8384(10)	6053(10)	1052(7)	58(4)
C(52)	7996(9)	6086(13)	488(8)	63(5)
C(53)	7217(15)	6120(20)	549(13)	126(12)
C(54)	8166(15)	5295(16)	137(10)	96(8)
C(55)	7851(10)	6827(14)	1848(8)	70(6)
C(56)	8122(11)	6574(13)	2448(8)	68(5)
C(57)	7510(14)	6521(19)	2835(12)	116(10)
C(58)	8523(13)	5777(15)	2475(9)	84(7)
S(11)	9112(2)	10122(3)	2239(2)	47(1)
C(12)	9390(7)	11177(11)	2165(7)	48(4)
N(13)	9835(7)	11470(8)	1800(6)	51(3)
C(14)	10023(7)	11268(11)	1392(7)	46(3)
O(15)	9838(5)	10503(7)	1065(4)	46(2)
C(141)	10635(7)	11622(10)	1129(6)	46(3)
C(142)	10796(9)	12404(11)	1328(7)	54(4)
C(143)	11344(9)	12858(13)	1126(8)	60(4)
C(144)	8390(7)	8348(10)	-545(6)	44(3)
C(145)	8222(8)	7535(12)	-755(7)	54(4)
C(146)	11036(8)	11220(11)	721(7)	51(4)

N(16)	9169(7)	11749(9)	2535(5)	49(3)
C(151)	8713(8)	11560(12)	2985(7)	55(4)
C(152)	9066(9)	11483(13)	3584(7)	59(4)
C(153)	9594(10)	10798(14)	3612(8)	72(6)
C(154)	8530(11)	11329(13)	4011(8)	70(5)
C(155)	9427(10)	12619(10)	2500(7)	56(4)
C(156)	9094(9)	13151(11)	2020(6)	54(4)
C(157)	8344(10)	13296(12)	2097(8)	65(5)
C(158)	9447(11)	13974(12)	1982(8)	70(5)
Re(2)	7764(1)	10197(1)	669(1)	48(1)
O(30)	7069(6)	10541(10)	294(5)	73(4)
S(21)	7215(2)	9014(4)	1058(2)	63(1)
C(22)	7125(7)	7988(10)	606(6)	40(3)
N(23)	7414(6)	8312(10)	29(6)	55(4)
C(24)	7931(7)	8772(11)	-148(7)	48(4)
O(25)	8057(5)	9532(7)	-41(5)	50(3)
N(26)	6345(8)	8250(15)	317(8)	89(6)
C(251)	6127(12)	7910(20)	-222(12)	108(10)
C(252)	6063(17)	8610(20)	-723(11)	112(9)
C(253)	5560(20)	9390(20)	-550(20)	180(20)
C(254)	5870(20)	8190(30)	-1278(14)	180(20)
C(255)	5819(10)	8534(19)	742(11)	91(8)
C(256)	5834(15)	7870(20)	1212(13)	108(10)
C(257)	5430(20)	8220(30)	1690(20)	190(20)
C(258)	5574(14)	7060(20)	1031(14)	118(11)
S(31)	7552(2)	11019(3)	1490(2)	61(1)
C(32)	7494(7)	11980(10)	1121(6)	43(3)
N(33)	8059(8)	12282(9)	830(6)	61(4)
C(34)	8417(6)	11927(12)	536(6)	46(4)
O(35)	8342(5)	11135(7)	312(4)	46(2)
N(36)	6946(7)	12435(10)	1101(6)	56(3)
C(351)	6349(8)	12202(14)	1472(8)	64(5)
C(352)	6478(10)	12510(17)	2075(9)	79(7)
C(353)	5897(15)	12150(30)	2441(15)	166(18)
C(354)	6509(15)	13460(20)	2115(14)	138(14)
C(355)	6849(9)	13178(15)	731(9)	72(6)
C(356)	6692(11)	12892(18)	117(10)	90(7)
C(357)	6694(16)	13610(20)	-265(12)	127(12)
C(358)	6056(11)	12350(19)	44(11)	96(8)

C(61)	7349(19)	9090(30)	2648(19)	76(12)
S(62)	7408(4)	9609(6)	3289(4)	55(2)
C(63)	6973(18)	10520(20)	3080(20)	69(11)
0(64)	6964(16)	9160(20)	3668(14)	98(11)
C(65)	6996(14)	730(20)	8910(13)	53(9)
S(66)	7524(4)	1595(5)	8932(3)	43(2)
C(67)	8299(11)	1042(19)	8864(13)	39(6)
O(68)	7382(10)	2099(15)	8390(10)	55(6)
O(69B)	8735(8)	1515(14)	1423(8)	76(5)
O(69A)	5030(20)	4230(30)	955(19)	52(10)

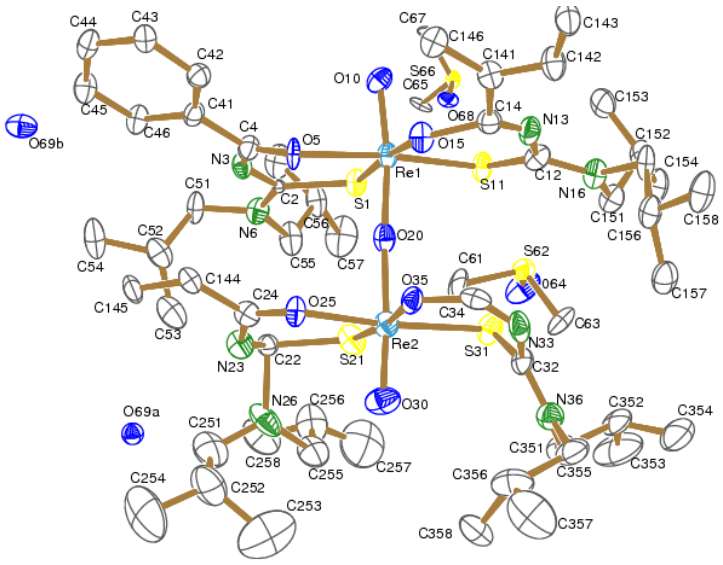

Figure 16 Ellipsoid plot (50% probability) of the asymmetric unit of [({ReOphth(*i*-Bu_2tu)$_2$}$_2$O)$_2$]·DMSO·H_2O.

17 [ReCl(OMe){dpic(*i*-Bu_2tu)$_2$}], (13)

Table 17.1 Crystal data and structure refinement for [ReCl(OMe){dpic(*i*-Bu_2tu)$_2$}].

Empirical formula	$C_{26}H_{42}ClN_5O_3ReS_2$	
Formula weight	758.42	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.862(3) Å	α= 90°
	b = 11.660(2) Å	β= 96.86(2)°
	c = 24.931(7) Å	γ = 90°
Volume	3423.4(14) Å^3	
Z	4	
Density (calculated)	1.472 g/cm^3	
Absorption coefficient	3.781 mm^{-1}	
F(000)	1524	
Crystal description	Needle	
Crystal color	Black-blue	
Crystal size	0.500 x 0.200 x 0.050 mm^3	
Theta range for data collection	1.65 to 29.52	
Index ranges	-16<=h<=13, -15<=k<=13, -34<=l<=34	
Reflections collected	31318	
Independent reflections	9276 [R(int) = 0.5233]	
Completeness to theta = 29.52°	97.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.6201 and 0.5349	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9276 / 0 / 208	
Goodness-of-fit on F^2	0.885	
Final R indices [I>2sigma(I)]	R1 = 0.1419, wR2 = 0.2768	
R indices (all data)	R1 = 0.4324, wR2 = 0.4267	
Largest diff. peak and hole	1.953 and -2.917 e.Å^{-3}	

Table 17.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReCl(OMe){dpic(*i*-Bu_2tu)$_2$}]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	3144(1)	8794(1)	8559(1)	54(1)
Cl	1296(8)	7981(9)	8539(5)	76(3)
O(10)	4650(19)	9544(18)	8606(8)	49(6)
C(10)	5180(40)	9630(40)	8227(17)	72(12)
S(1)	2630(10)	9370(9)	7614(4)	74(3)
C(2)	1890(30)	10480(30)	7907(16)	61(10)
N(3)	2270(30)	10400(30)	8465(14)	68(9)
C(4)	2430(30)	11260(40)	8889(15)	72(10)
O(5)	2270(20)	12230(20)	8869(14)	95(11)
N(6)	1269(18)	11210(30)	7626(11)	51(7)
C(41)	1090(30)	11240(40)	7052(15)	77(11)
C(42)	1550(70)	12200(70)	6750(30)	170(30)
C(43)	1430(60)	11840(70)	6150(30)	190(30)
C(44)	2760(90)	12220(90)	6920(40)	280(50)
C(45)	480(40)	11860(40)	7911(18)	81(13)
C(46)	-490(50)	11410(60)	8140(30)	140(20)
C(47)	-1100(50)	10640(50)	7710(20)	140(20)
C(48)	-1050(60)	12380(60)	8480(30)	170(30)
S(11)	3813(8)	7058(8)	8111(4)	56(2)
C(12)	3850(20)	6470(20)	8749(12)	38(8)
N(13)	3830(30)	7460(20)	9104(12)	71(10)
C(14)	4110(30)	7640(30)	9613(16)	58(10)
O(15)	4630(20)	7010(20)	9945(9)	71(8)
C(31)	2910(30)	10590(30)	9397(16)	65(11)
C(32)	3070(30)	11080(40)	9876(14)	67(10)
C(33)	3450(40)	10490(40)	10340(19)	86(13)
C(34)	3840(40)	9320(40)	10255(17)	77(12)
C(35)	3630(30)	8830(40)	9748(14)	66(9)
N(36)	3190(20)	9410(20)	9337(10)	42(7)
N(16)	3790(40)	5450(20)	8916(13)	97(14)
C(52)	4960(30)	3880(30)	8719(14)	58(9)
C(53)	6180(50)	4480(60)	8780(30)	160(20)
C(54)	5080(40)	2930(40)	8340(20)	105(17)
C(51)	4030(30)	4550(30)	8534(15)	69(11)

C(55)	3190(40)	5070(40)	9425(17)	83(13)
C(56)	2040(40)	5330(40)	9463(17)	83(13)
C(57)	1730(40)	4950(40)	10013(18)	94(15)
C(58)	1260(40)	4940(40)	9039(17)	83(13)

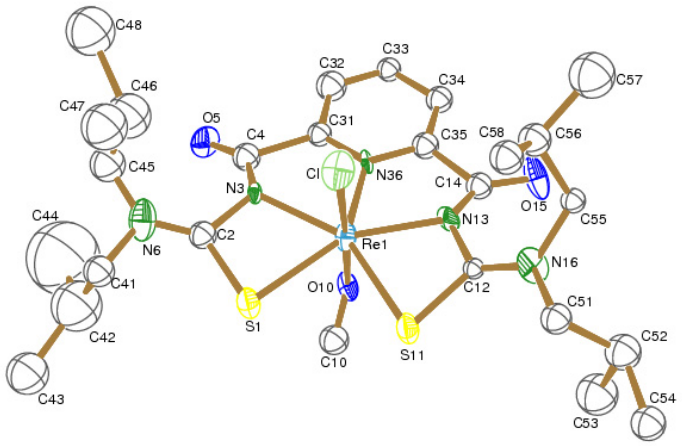

Figure 17 Ellipsoid plot (30% probability) of [ReCl(OMe){dpic(*i*-Bu_2tu)$_2$}]. All carbon atoms were refined with isotropical thermal parameters.

18 H_2L^3

Table 18.1 Crystal data and structure refinement for $H_2L^3 \cdot$ MeOH.

Empirical formula	$C_{20}H_{25}N_3O_3S$	
Formula weight	387.49	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 21.356(3) Å	α= 90°
	b = 10.809(1) Å	β= 102.37(1)°
	c = 18.663(2) Å	γ = 90°
Volume	4208.1(8) $Å^3$	
Z	8	
Density (calculated)	1.223 g/cm^3	
Absorption coefficient	0.178 mm^{-1}	
F(000)	1648	
Crystal description	Block	
Crystal color	Colorless	
Crystal size	0.15 x 0.1 x 0.1 mm^3	
Theta range for data collection	1.95 to 29.30	
Index ranges	-29<=h<=22, -14<=k<=13, -25<=l<=25	
Reflections collected	14200	
Independent reflections	5641 [R(int) = 0.1247]	
Completeness to theta = 29.30°	97.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5641 / 0 / 237	
Goodness-of-fit on F^2	0.897	
Final R indices [I>2sigma(I)]	R1 = 0.0653, wR2 = 0.1353	
R indices (all data)	R1 = 0.1857, wR2 = 0.1978	
Extinction coefficient	0.0052(6)	
Largest diff. peak and hole	0.506 and -0.335 $e.Å^{-3}$	

Table 18.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $H_2L^3 \cdot$ MeOH. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	1781(1)	6231(1)	2041(1)	54(1)
C(2)	1578(2)	5554(4)	1211(2)	44(1)
N(3)	1288(1)	6234(3)	604(1)	49(1)
C(4)	722(2)	6705(3)	521(2)	44(1)
N(5)	511(1)	7582(3)	0(1)	46(1)
C(31)	233(2)	6318(3)	941(2)	44(1)
C(32)	-152(2)	7172(4)	1192(2)	58(1)
C(33)	-592(2)	6793(5)	1589(2)	70(1)
C(34)	-670(2)	5558(5)	1718(2)	69(1)
C(35)	-302(2)	4697(4)	1462(2)	60(1)
C(36)	149(2)	5072(4)	1075(2)	50(1)
N(6)	1742(1)	4408(3)	1073(1)	51(1)
C(41)	2102(2)	3606(4)	1660(2)	61(1)
C(42)	1661(2)	2887(4)	2033(2)	79(1)
C(43)	1573(2)	3861(4)	334(2)	60(1)
C(44)	2085(3)	4083(5)	-96(3)	88(2)
C(51)	824(2)	8151(3)	-502(2)	43(1)
C(52)	1463(2)	7936(4)	-518(2)	52(1)
C(53)	1739(2)	8510(4)	-1027(2)	59(1)
C(54)	1392(2)	9301(4)	-1539(2)	63(1)
C(55)	759(2)	9520(4)	-1532(2)	55(1)
C(56)	465(2)	8969(3)	-1023(2)	45(1)
C(57)	-220(2)	9257(3)	-1046(2)	49(1)
O(57)	-472(1)	9998(3)	-1585(1)	66(1)
O(58)	-527(1)	8853(3)	-615(1)	67(1)
O(62)	-1605(2)	10884(3)	-1699(2)	75(1)
C(61)	-2041(2)	10501(5)	-1285(2)	79(1)

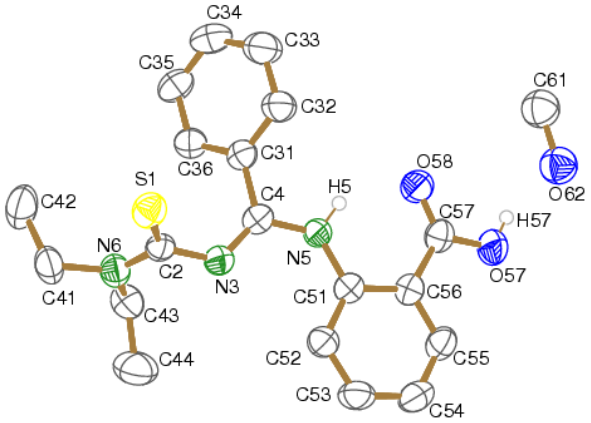

Figure 18 Ellipsoid plot (50% probability) of $H_2L^3 \cdot MeOH$.

19 H_2L^{5a}

Table 19.1 Crystal data and structure refinement for H_2L^{5a}.

Empirical formula	$C_{15}H_{23}N_5S_2$	
Formula weight	337.52	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.603(1) Å	α= 63.66(1)°
	b = 10.226(1) Å	β= 65.26(1)°
	c = 10.978(1) Å	γ = 83.92(1)°
Volume	873.27(15) $Å^3$	
Z	2	
Density (calculated)	1.284 g/cm^3	
Absorption coefficient	0.309 mm^{-1}	
F(000)	360	
Crystal description	Block	
Crystal color	Colorless	
Crystal size	0.3 x 0.2 x 0.1 mm^3	
Theta range for data collection	2.78 to 29.29	
Index ranges	-13<=h<=13, -13<=k<=13, -14<=l<=12	
Reflections collected	8476	
Independent reflections	4488 [R(int) = 0.0298]	
Completeness to theta = 29.29°	94.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4488 / 0 / 207	
Goodness-of-fit on F^2	0.855	
Final R indices [I>2sigma(I)]	R1 = 0.0399, wR2 = 0.0979	
R indices (all data)	R1 = 0.0689, wR2 = 0.1084	
Largest diff. peak and hole	0.237 and -0.219 $e.Å^{-3}$	

Table 19.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for H_2L^{5a}. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	4467(1)	9658(1)	3102(1)	43(1)
C(2)	4201(2)	7849(2)	3575(2)	32(1)
N(3)	4944(2)	7293(2)	2567(2)	34(1)
C(4)	4922(2)	7714(2)	1286(2)	30(1)
N(5)	3881(2)	8500(2)	805(2)	34(1)
C(31)	6135(2)	7241(2)	215(2)	32(1)
C(32)	5808(2)	6824(2)	-682(2)	40(1)
C(33)	6934(2)	6286(2)	-1584(3)	53(1)
C(34)	8379(2)	6170(2)	-1591(3)	57(1)
C(35)	8719(2)	6606(2)	-722(3)	53(1)
C(36)	7603(2)	7143(2)	189(2)	41(1)
N(6)	3396(1)	6846(2)	4968(2)	36(1)
C(41)	2725(2)	7204(3)	6236(2)	50(1)
C(42)	3784(3)	6997(3)	6990(3)	60(1)
C(43)	3072(2)	5329(2)	5286(2)	44(1)
C(44)	1716(2)	5165(2)	4976(3)	52(1)
N(7)	2559(2)	8818(2)	1750(2)	35(1)
C(8)	1156(2)	8525(2)	1825(2)	33(1)
S(9)	1013(1)	7699(1)	863(1)	50(1)
N(51)	-36(2)	8921(2)	2744(2)	40(1)
C(52)	133(2)	9441(3)	3714(3)	54(1)
C(53)	-1569(2)	8922(3)	2761(3)	55(1)

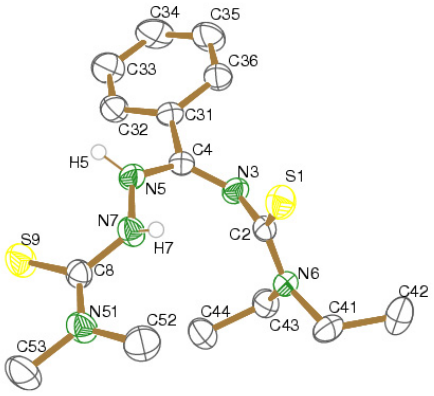

Figure 19 Ellipsoid plot (50% probability) of H_2L^{5a}.

20 H_2L^{5b}

Table 20.1 Crystal data and structure refinement for H_2L^{5b}.

Empirical formula	$C_{17}H_{25}N_5S_2$	
Formula weight	363.54	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 15.268(1) Å	α= 90°
	b = 13.408(4) Å	β= 99.55(7)°
	c = 18.941(1) Å	γ = 90°
Volume	3824(4) $Å^3$	
Z	8	
Density (calculated)	1.263 g/cm^3	
Absorption coefficient	0.287 mm^{-1}	
F(000)	1552	
Crystal description	Block	
Crystal color	Colorless	
Crystal size	0.45 x 0.35 x 0.30 mm^3	
Theta range for data collection	2.66 to 29.40	
Index ranges	-20<=h<=20, -18<=k<=18, -18<=l<=26	
Reflections collected	28246	
Independent reflections	10304 [R(int) = 0.1063]	
Completeness to theta = 29.40°	97.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10304 / 0 / 433	
Goodness-of-fit on F^2	0.920	
Final R indices [I>2sigma(I)]	R1 = 0.0648, wR2 = 0.1189	
R indices (all data)	R1 = 0.1546, wR2 = 0.1458	
Largest diff. peak and hole	0.388 and -0.334 $e.Å^{-3}$	

Table 20.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for H_2L^{5b}. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	1963(1)	3410(1)	2915(1)	41(1)
C(2)	2945(2)	2818(2)	2932(2)	31(1)
N(3)	2991(2)	1804(2)	3027(1)	31(1)
C(4)	2281(2)	1209(2)	3185(2)	28(1)
N(5)	1605(2)	940(2)	2726(1)	32(1)
N(7)	1549(2)	1334(2)	2051(1)	33(1)
C(8)	860(2)	1084(2)	1529(2)	30(1)
S(9)	182(1)	114(1)	1594(1)	36(1)
C(31)	2364(2)	806(2)	3920(2)	31(1)
C(32)	1862(2)	-21(2)	4057(2)	43(1)
C(33)	1966(3)	-422(3)	4739(2)	58(1)
C(34)	2552(3)	0(4)	5294(2)	69(1)
C(35)	3022(3)	832(4)	5165(2)	70(1)
C(36)	2937(2)	1231(3)	4480(2)	50(1)
N(6)	3698(2)	3277(2)	2847(2)	39(1)
C(41)	4533(2)	2768(2)	2809(2)	44(1)
C(42)	4581(3)	2391(3)	2075(3)	65(1)
C(43)	3717(3)	4353(2)	2695(2)	59(1)
C(44)	4009(5)	4950(4)	3363(3)	113(2)
N(51)	778(2)	1669(2)	947(1)	33(1)
C(52)	84(3)	1510(2)	323(2)	45(1)
C(53)	303(4)	2276(3)	-216(2)	75(2)
C(54)	793(3)	3061(3)	205(2)	72(1)
C(55)	1324(3)	2553(2)	854(2)	42(1)
S(21)	2963(1)	6991(1)	1774(1)	45(1)
C(22)	2000(2)	7529(2)	1890(2)	30(1)
N(23)	1941(2)	8550(2)	1886(1)	30(1)
C(24)	2669(2)	9180(2)	1796(2)	28(1)
N(25)	3305(2)	9398(2)	2303(1)	31(1)
N(27)	3305(2)	8923(2)	2940(1)	34(1)
C(28)	3931(2)	9128(2)	3518(2)	30(1)
S(29)	4644(1)	10091(1)	3547(1)	36(1)
C(231)	2660(2)	9672(2)	1101(2)	30(1)
C(232)	3341(3)	10323(3)	1009(2)	49(1)

C(233)	3325(3)	10819(3)	368(2)	62(1)
C(234)	2635(3)	10670(3)	-184(2)	53(1)
C(235)	1968(3)	10030(3)	-111(2)	50(1)
C(236)	1976(2)	9528(3)	535(2)	40(1)
N(26)	1271(2)	7028(2)	2001(1)	32(1)
C(241)	480(2)	7513(2)	2191(2)	38(1)
C(242)	615(3)	7828(3)	2967(2)	55(1)
C(243)	1271(2)	5934(2)	2058(2)	42(1)
C(244)	702(3)	5444(3)	1437(2)	63(1)
N(251)	3930(2)	8495(2)	4065(1)	35(1)
C(252)	3357(3)	7614(2)	4053(2)	46(1)
C(253)	3730(5)	7092(4)	4741(3)	131(3)
C(254)	4332(4)	7703(3)	5163(3)	76(2)
C(255)	4528(3)	8603(3)	4750(2)	49(1)

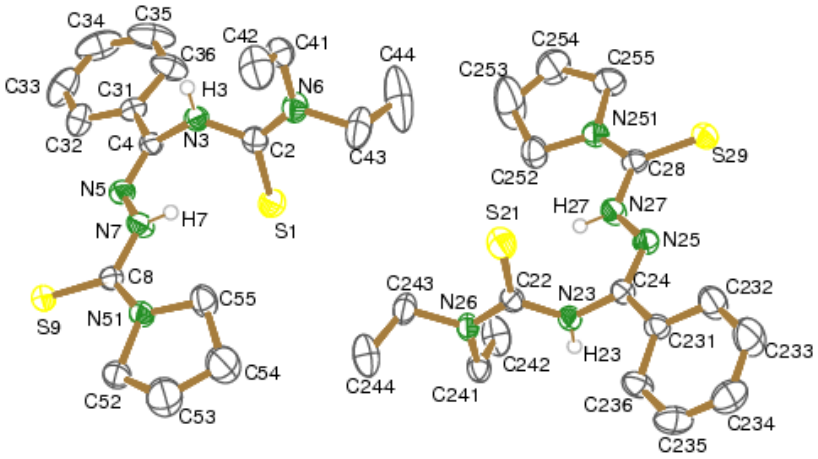

Figure 20 Ellipsoid plot (50% probability) of two crystallographically independent species of H_2L[5b].

21 [ReOCl(L^{1b})], (14b)

Table 21.1 Crystal data and structure refinement for [ReOCl(L^{1b})].

Empirical formula	$C_{18}H_{17}ClN_3O_3ReS$	
Formula weight	577.06	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 10.310(1) Å	α= 90°
	b = 12.389(1) Å	β= 103.25(1)°
	c = 15.295(1) Å	γ = 90°
Volume	1901.6(3) $Å^3$	
Z	4	
Density (calculated)	2.016 g/cm^3	
Absorption coefficient	6.665 mm^{-1}	
F(000)	1112	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.240 x 0.177 x 0.110 mm^3	
Theta range for data collection	2.14 to 29.32	
Index ranges	-14<=h<=11, -17<=k<=16, -20<=l<=20	
Reflections collected	16231	
Independent reflections	5099 [R(int) = 0.0828]	
Completeness to theta = 29.32°	98.1 %	
Absorption correction	Integration	
Max. and min. transmission	0.7656 and 0.3515	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5099 / 0 / 244	
Goodness-of-fit on F^2	1.097	
Final R indices [I>2sigma(I)]	R1 = 0.0476, wR2 = 0.1178	
R indices (all data)	R1 = 0.0736, wR2 = 0.1459	
Largest diff. peak and hole	1.185 and -1.939 $e.Å^{-3}$	

Table 21.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReOCl(L^{1b})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	3536(1)	6450(1)	1295(1)	24(1)
O(10)	4132(8)	6074(6)	417(4)	38(2)
Cl	3445(3)	8339(2)	1206(2)	45(1)
S(1)	1261(2)	6508(2)	862(1)	30(1)
C(2)	662(9)	5259(7)	1201(6)	29(2)
N(3)	1397(7)	4365(6)	1300(5)	26(1)
C(4)	2689(8)	4285(7)	1608(5)	24(2)
N(5)	3480(6)	5113(5)	2017(4)	21(1)
C(31)	3271(8)	3230(7)	1477(5)	22(1)
C(32)	2569(10)	2277(7)	1506(6)	29(2)
C(33)	3111(10)	1287(7)	1325(6)	32(2)
C(34)	4309(12)	1270(7)	1079(7)	38(2)
C(35)	5003(12)	2223(8)	1021(7)	40(2)
C(36)	4524(10)	3195(8)	1259(6)	33(2)
N(6)	-593(9)	5209(7)	1291(6)	41(2)
O(43)	-3061(8)	5163(8)	1731(7)	59(2)
C(41)	-1106(10)	4184(9)	1556(8)	42(2)
C(42)	-2018(16)	4439(11)	2161(10)	63(3)
C(44)	-2452(16)	6179(11)	1599(11)	63(4)
C(45)	-1570(12)	6032(11)	943(9)	51(3)
C(51)	4560(10)	5001(6)	2779(5)	28(2)
C(52)	4813(12)	4120(7)	3371(6)	37(2)
C(53)	5893(12)	4208(8)	4109(7)	43(2)
C(54)	6588(12)	5148(8)	4290(7)	47(3)
C(55)	6268(10)	6040(8)	3737(7)	38(2)
C(56)	5288(9)	5947(7)	2962(6)	28(2)
O(57)	4895(6)	6774(5)	2384(4)	27(1)

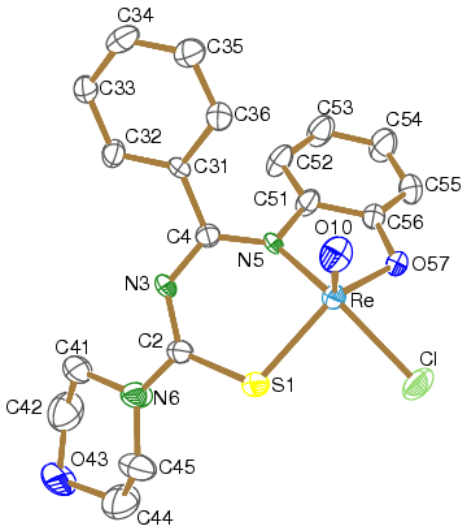

Figure 21 Ellipsoid plot (50% probability) of [ReOCl(L^{1b})].

22 [TcOCl(L^{1a})], (15a)

Table 22.1 Crystal data and structure refinement for [TcOCl(L^{1a})].

Empirical formula	$C_{18}H_{19}ClN_3O_2STc$	
Formula weight	474.87	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 11.406(1) Å	α= 90°
	b = 12.878(1) Å	β= 93.06(1)°
	c = 13.007(1) Å	γ = 90°
Volume	1907.8(3) $Å^3$	
Z	4	
Density (calculated)	1.653 g/cm^3	
Absorption coefficient	1.022 mm^{-1}	
F(000)	960	
Crystal description	Plate	
Crystal color	Black	
Crystal size	0.200 x 0.163 x 0.090 mm^3	
Theta range for data collection	2.23 to 29.22	
Index ranges	-15<=h<=14, -17<=k<=15, -14<=l<=17	
Reflections collected	9486	
Independent reflections	5054 [R(int) = 0.0766]	
Completeness to theta = 29.22°	97.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5054 / 0 / 235	
Goodness-of-fit on F^2	0.865	
Final R indices [I>2sigma(I)]	R1 = 0.0497, wR2 = 0.1271	
R indices (all data)	R1 = 0.1096, wR2 = 0.1960	
Largest diff. peak and hole	1.069 and -1.848 $e.Å^{-3}$	

Table 22.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcOCl(L^{1a})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	1983(1)	1569(1)	4441(1)	26(1)
O(10)	1662(4)	1096(4)	5565(4)	37(1)
Cl	1984(2)	3385(1)	4625(2)	36(1)
S(1)	3968(1)	1819(1)	4356(2)	33(1)
C(2)	4539(5)	671(6)	3847(6)	33(2)
N(3)	4001(4)	-258(5)	3892(5)	29(1)
C(4)	2877(5)	-426(5)	3774(6)	28(1)
N(5)	2053(4)	336(4)	3523(4)	21(1)
C(31)	2490(5)	-1502(5)	3957(5)	23(1)
C(32)	3261(6)	-2326(5)	3803(6)	30(2)
C(33)	2963(6)	-3324(5)	4062(6)	35(2)
C(34)	1891(7)	-3507(6)	4509(6)	37(2)
C(35)	1133(6)	-2689(6)	4668(6)	32(2)
C(36)	1421(5)	-1703(5)	4385(5)	28(1)
N(6)	5584(5)	696(5)	3458(5)	33(1)
C(41)	6058(6)	-253(6)	2958(7)	42(2)
C(42)	5515(8)	-453(8)	1927(8)	55(2)
C(43)	6337(6)	1654(6)	3469(6)	36(2)
C(44)	6251(7)	2190(7)	2422(8)	47(2)
C(51)	1050(5)	171(5)	2829(5)	24(1)
C(52)	859(6)	-603(5)	2135(5)	26(1)
C(53)	-122(7)	-532(6)	1460(7)	41(2)
C(54)	-883(6)	292(6)	1493(7)	39(2)
C(55)	-687(6)	1094(6)	2190(6)	31(2)
C(56)	301(5)	1029(5)	2861(6)	29(1)
O(57)	621(4)	1830(4)	3515(4)	31(1)

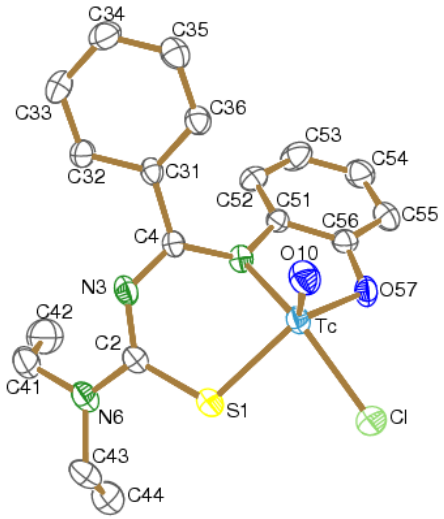

Figure 22 Ellipsoid plot (50% probability) of [TcOCl(L^{1a})].

23 [ReO(OMe)(L^{1a})], (16a)

Table 23.1 Crystal data and structure refinement for [ReO(OMe)(L^{1a})].

Empirical formula	$C_{19}H_{22}N_3O_3ReS$	
Formula weight	558.66	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 8.667(1) Å	α= 90°
	b = 13.159(1) Å	β= 91.90(1)°
	c = 17.152(1) Å	γ = 90°
Volume	1955.3(3) $Å^3$	
Z	4	
Density (calculated)	1.898 g/cm^3	
Absorption coefficient	6.346 mm^{-1}	
F(000)	1088	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.100 mm^3	
Theta range for data collection	1.95 to 29.30	
Index ranges	-11<=h<=11, -18<=k<=16, -22<=l<=23	
Reflections collected	13201	
Independent reflections	5236 [R(int) = 0.0600]	
Completeness to theta = 29.30°	98.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.5860 and 0.1736	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5236 / 0 / 245	
Goodness-of-fit on F^2	0.929	
Final R indices [I>2sigma(I)]	R1 = 0.0386, wR2 = 0.1129	
R indices (all data)	R1 = 0.0577, wR2 = 0.1493	
Extinction coefficient	0.0061(7)	
Largest diff. peak and hole	1.989 and -1.546 $e.Å^{-3}$	

Table 23.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(OMe)(L^{1a})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	6143(1)	1575(1)	8654(1)	27(1)
O(10)	5155(6)	1902(5)	9451(3)	37(1)
O(20)	6109(7)	59(5)	8578(3)	44(2)
C(20)	6930(11)	-529(8)	8977(7)	56(3)
S(1)	4436(2)	1429(1)	7613(1)	33(1)
C(2)	4417(7)	2601(5)	7130(4)	27(1)
N(3)	4886(7)	3477(4)	7449(3)	29(1)
C(4)	6021(8)	3606(5)	7979(4)	28(1)
N(5)	7021(6)	2859(4)	8205(3)	24(1)
C(31)	6094(7)	4640(5)	8333(4)	27(1)
C(32)	5666(9)	5487(6)	7909(4)	34(2)
C(33)	5701(12)	6422(6)	8260(6)	45(2)
C(34)	6065(11)	6516(6)	9065(6)	42(2)
C(35)	6464(9)	5659(7)	9483(5)	40(2)
C(36)	6507(8)	4714(6)	9120(4)	34(1)
N(6)	3842(6)	2638(5)	6408(4)	34(1)
C(41)	3790(10)	3608(7)	5955(5)	40(2)
C(42)	2267(12)	4149(8)	6082(6)	57(3)
C(43)	3210(9)	1737(6)	5991(5)	38(2)
C(44)	4487(10)	1170(7)	5575(5)	43(2)
C(51)	8625(7)	3033(5)	8377(4)	25(1)
C(52)	9505(8)	3849(6)	8157(4)	33(1)
C(53)	11088(8)	3828(7)	8348(5)	36(2)
C(54)	11745(9)	3013(7)	8714(5)	38(2)
C(55)	10872(8)	2179(6)	8921(5)	36(2)
C(56)	9285(8)	2194(6)	8756(4)	30(1)
O(57)	8358(6)	1381(4)	8881(3)	31(1)

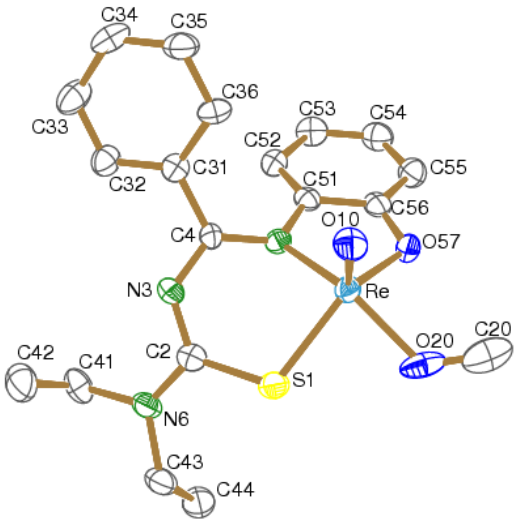

Figure 23 Ellipsoid plot (50% probability) of [ReO(OMe)(L^{1a})].

24 [{ReO(L^{1a})}$_2$O], (17a)

Table 24.1 Crystal data and structure refinement for [{ReO(L^{1a})}$_2$O]·1/2CH_2Cl_2.

Empirical formula	$C_{36.50}H_{39}ClN_6O_5Re_2S_2$	
Formula weight	1113.71	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 14.923(1) Å	α= 90°
	b = 18.166(1) Å	β= 97.24(1)°
	c = 15.381(1) Å	γ = 90°
Volume	4136.3(4) Å^3	
Z	4	
Density (calculated)	1.788 g/cm^3	
Absorption coefficient	6.060 mm^{-1}	
F(000)	2156	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.140 x 0.020 mm^3	
Theta range for data collection	2.75 to 29.31	
Index ranges	-20<=h<=20, -24<=k<=21, -21<=l<=14	
Reflections collected	24313	
Independent reflections	10938 [R(int) = 0.1784]	
Completeness to theta = 29.31°	96.5 %	
Absorption correction	Integration	
Max. and min. transmission	0.5224 and 0.2789	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10938 / 0 / 487	
Goodness-of-fit on F^2	0.837	
Final R indices [I>2sigma(I)]	R1 = 0.0767, wR2 = 0.1098	
R indices (all data)	R1 = 0.2296, wR2 = 0.1530	
Largest diff. peak and hole	1.003 and -1.053 e.Å^{-3}	

Table 24.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(L^{1a})}$_2$O]·1/2CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	435(1)	1413(1)	1844(1)	45(1)
O(10)	600(8)	1086(6)	867(8)	72(4)
O(20)	-213(6)	909(5)	2623(7)	52(3)
S(1)	1712(3)	970(2)	2695(3)	60(1)
C(2)	2405(9)	1743(8)	2936(11)	45(4)
N(3)	2396(8)	2335(7)	2448(10)	54(4)
C(4)	1692(9)	2637(8)	1936(12)	48(4)
N(5)	796(8)	2455(6)	1987(9)	45(3)
C(31)	1940(12)	3196(9)	1290(14)	59(5)
C(32)	1456(11)	3172(9)	455(13)	58(5)
C(33)	1707(16)	3647(13)	-193(15)	92(7)
C(34)	2450(20)	4133(11)	60(20)	107(9)
C(35)	2917(15)	4111(13)	900(20)	97(7)
C(36)	2679(12)	3654(9)	1534(14)	73(5)
N(6)	3052(10)	1690(8)	3611(12)	74(5)
C(41)	3208(12)	1029(11)	4154(14)	78(6)
C(42)	3881(15)	545(12)	3832(17)	104(8)
C(43)	3970(30)	2296(18)	3770(30)	210(20)
C(44)	3540(30)	2630(30)	4270(30)	260(30)
C(51)	91(9)	2998(9)	1837(11)	49(4)
C(52)	175(11)	3759(8)	1894(12)	54(4)
C(53)	-617(12)	4186(8)	1728(13)	63(5)
C(54)	-1450(10)	3863(8)	1546(11)	51(4)
C(55)	-1521(11)	3114(8)	1492(11)	51(4)
C(56)	-735(10)	2673(7)	1647(11)	43(4)
O(57)	-781(6)	1929(5)	1616(7)	49(3)
Re(2)	-1559(1)	956(1)	2423(1)	47(1)
O(110)	-1961(7)	257(5)	2961(9)	65(4)
S(11)	-1499(3)	1884(2)	3444(3)	54(1)
C(12)	-2461(11)	2428(8)	3157(10)	49(4)
N(13)	-3168(8)	2206(7)	2633(10)	54(4)
C(14)	-3295(9)	1705(8)	2014(11)	41(4)
N(15)	-2613(7)	1374(7)	1652(9)	48(3)
C(131)	-4256(8)	1477(9)	1749(11)	51(4)

C(132)	-4942(11)	1995(11)	1722(13)	75(6)
C(133)	-5827(11)	1798(14)	1474(12)	74(6)
C(134)	-6047(12)	1056(16)	1366(14)	93(8)
C(135)	-5357(14)	556(13)	1449(16)	96(8)
C(136)	-4473(11)	753(9)	1634(12)	58(5)
N(16)	-2482(9)	3089(8)	3492(10)	63(4)
C(141)	-3191(13)	3643(10)	3156(15)	77(5)
C(142)	-4001(15)	3603(13)	3648(16)	112(8)
C(143)	-1822(13)	3346(10)	4247(14)	79(6)
C(144)	-998(15)	3725(13)	3933(18)	127(10)
C(151)	-2709(8)	1124(7)	753(11)	42(4)
C(152)	-3343(10)	1340(9)	62(12)	56(4)
C(153)	-3292(12)	1072(10)	-775(15)	73(6)
C(154)	-2619(12)	637(11)	-894(14)	79(6)
C(155)	-1916(11)	411(9)	-221(14)	63(5)
C(156)	-1986(10)	677(8)	585(13)	56(5)
O(157)	-1347(7)	514(5)	1282(8)	56(3)
C(61)	376(17)	1119(14)	4750(20)	44(8)
Cl(63)	962(7)	1934(5)	4687(8)	83(3)
Cl(62)	764(7)	642(5)	5680(7)	92(4)

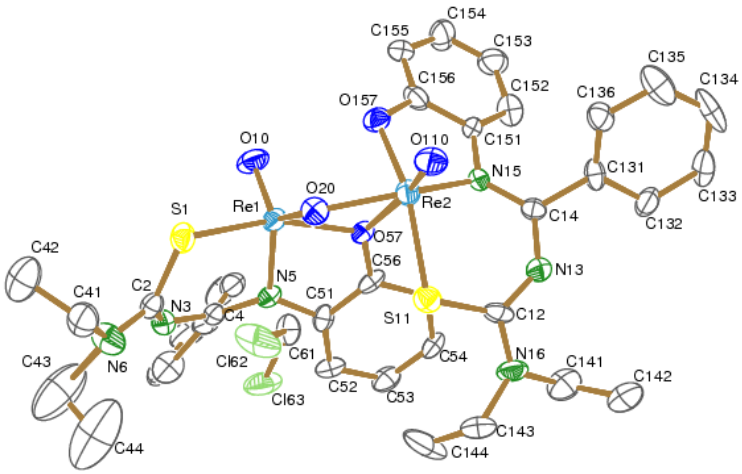

Figure 24 Ellipsoid plot (50% probability) of [{ReO(L^{1a})}$_2$O]·1/2CH_2Cl_2.

25 [{ReO(L^{1b})}$_2$S], (18b)

Table 25.1 Crystal data and structure refinement for [{ReO(L^{1b})}$_2$S]·2CH_2Cl_2.

Empirical formula	$C_{38}H_{38}Cl_4N_6O_6Re_2S_3$	
Formula weight	1285.12	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 10.273(1) Å	α= 90°
	b = 16.045(1) Å	β= 96.57(1)°
	c = 13.202(1) Å	γ = 90°
Volume	2161.7(3) Å^3	
Z	2	
Density (calculated)	1.974 g/cm^3	
Absorption coefficient	6.040 mm^{-1}	
F(000)	1244	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.100 mm^3	
Theta range for data collection	2.37 to 29.24	
Index ranges	-14<=h<=14, -21<=k<=22, -18<=l<=18	
Reflections collected	19178	
Independent reflections	11454 [R(int) = 0.0900]	
Completeness to theta = 29.24°	99.1 %	
Absorption correction	Integration	
Max. and min. transmission	0.5788 and 0.3587	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11454 / 1 / 532	
Goodness-of-fit on F^2	0.839	
Final R indices [I>2sigma(I)]	R1 = 0.0510, wR2 = 0.0713	
R indices (all data)	R1 = 0.1145, wR2 = 0.0837	
Absolute structure parameter	-0.006(9)	
Largest diff. peak and hole	1.832 and -1.126 e.Å^{-3}	

Table 25.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(L^{1b})}$_2$S]·2CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	4528(1)	2356(1)	5936(1)	34(1)
O(10)	4923(7)	1988(5)	4846(6)	54(2)
S(20)	6524(3)	2052(2)	7019(2)	43(1)
S(1)	3751(3)	1225(2)	6732(2)	44(1)
C(2)	2049(10)	1339(7)	6586(9)	36(3)
N(3)	1356(8)	1706(6)	5783(7)	35(2)
C(4)	1740(8)	2362(7)	5308(7)	31(2)
N(5)	2724(7)	2877(5)	5669(6)	30(2)
C(31)	1008(9)	2538(6)	4250(8)	38(3)
C(32)	-333(9)	2387(8)	4067(9)	54(3)
C(33)	-931(11)	2551(8)	3089(9)	58(3)
C(34)	-213(13)	2839(8)	2339(11)	61(4)
C(35)	1075(13)	2979(7)	2548(9)	54(3)
C(36)	1703(9)	2843(6)	3494(8)	39(3)
N(6)	1394(9)	997(5)	7281(7)	40(2)
C(42)	-20(10)	1045(7)	7270(9)	47(3)
C(43)	-594(13)	195(9)	7453(10)	54(4)
O(44)	15(9)	-164(6)	8397(7)	62(3)
C(45)	1382(14)	-234(8)	8358(10)	59(4)
C(46)	1972(13)	586(7)	8220(9)	53(3)
C(51)	2744(9)	3749(6)	5486(7)	26(2)
C(52)	1677(10)	4270(6)	5262(8)	32(2)
C(53)	1858(12)	5096(8)	5151(10)	48(3)
C(54)	3101(11)	5440(8)	5292(9)	44(3)
C(55)	4177(10)	4937(6)	5539(8)	34(3)
C(56)	3992(10)	4099(6)	5674(7)	33(2)
O(57)	4997(6)	3547(5)	5982(5)	37(2)
Re(2)	6252(1)	2655(1)	8546(1)	34(1)
O(110)	7034(8)	3572(5)	8651(6)	51(2)
S(11)	7712(3)	1653(2)	9198(2)	48(1)
C(12)	7421(10)	1448(7)	10442(8)	40(3)
N(13)	6771(8)	1992(6)	11009(7)	44(2)
C(14)	5777(8)	2471(6)	10676(7)	33(2)
N(15)	5151(6)	2451(5)	9698(5)	31(2)

C(131)	5357(10)	3096(7)	11394(8)	33(2)
C(132)	4932(11)	3858(8)	11070(9)	47(3)
C(133)	4595(12)	4470(7)	11738(10)	50(3)
C(134)	4661(11)	4280(8)	12761(10)	44(3)
C(135)	5084(11)	3523(8)	13086(9)	49(3)
C(136)	5467(10)	2919(7)	12443(7)	40(3)
N(16)	7887(10)	765(7)	10885(7)	55(3)
C(142)	7904(15)	545(9)	11978(10)	71(4)
C(143)	9320(15)	466(10)	12399(10)	78(5)
O(144)	9996(11)	-165(6)	11903(8)	67(3)
C(145)	9940(15)	29(8)	10855(10)	65(4)
C(146)	8582(14)	89(9)	10342(11)	66(4)
C(151)	3827(7)	2584(6)	9473(6)	26(2)
C(152)	2816(8)	2484(6)	10120(7)	37(3)
C(154)	1207(10)	2897(6)	8763(8)	37(3)
C(153)	1553(8)	2652(7)	9740(7)	37(2)
C(155)	2155(10)	3013(6)	8117(8)	36(3)
C(156)	3427(9)	2823(6)	8464(7)	29(2)
O(157)	4436(6)	2866(4)	7853(5)	33(2)
C(61)	-1910(12)	4055(8)	6260(10)	55(3)
Cl(62)	-818(3)	3253(2)	6394(3)	72(1)
Cl(63)	-1972(5)	4599(5)	5133(6)	151(3)
C(64)	6110(20)	5283(11)	8018(12)	114(8)
Cl(65)	5743(5)	5456(3)	9242(3)	90(1)
Cl(66)	6834(5)	6076(3)	7437(4)	99(1)

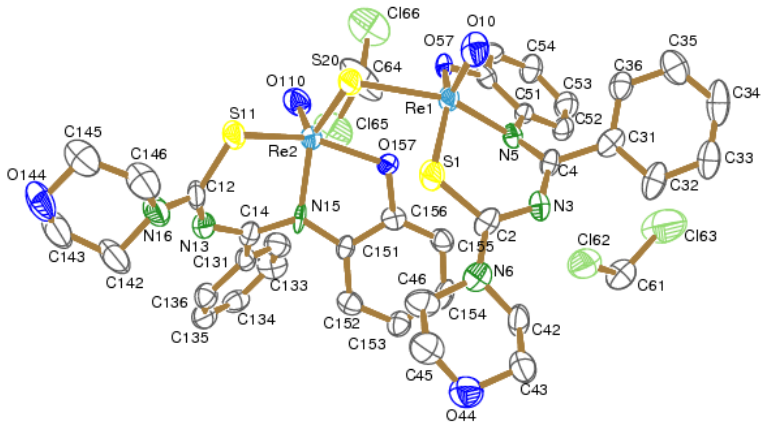

Figure 25 Ellipsoid plot (50% probability) of [{ReO(L^{1b})}$_2$S]·2CH_2Cl_2.

26 [ReO(L^{1a})(gly)], (19)

Table 26.1 Crystal data and structure refinement for [ReO(L^{1a})(gly)].

Empirical formula	$C_{20}H_{23}N_4O_4ReS$	
Formula weight	601.52	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 13.529(1) Å	α= 90°
	b = 14.421(1) Å	β= 91.11(1)°
	c = 10.886(1) Å	γ = 90°
Volume	2123.6(3) $Å^3$	
Z	4	
Density (calculated)	1.881 g/cm^3	
Absorption coefficient	5.855 mm^{-1}	
F(000)	1176	
Crystal description	Needle	
Crystal color	Red	
Crystal size	0.2 x 0.1 x 0.03 mm^3	
Theta range for data collection	2.06 to 26.75	
Index ranges	-17<=h<=12, -18<=k<=18, -13<=l<=13	
Reflections collected	12394	
Independent reflections	4451 [R(int) = 0.0568]	
Completeness to theta = 26.75°	98.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.6873 and 0.2196	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4451 / 0 / 295	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0305, wR2 = 0.0644	
R indices (all data)	R1 = 0.0521, wR2 = 0.0924	
Extinction coefficient	0.0051(3)	
Largest diff. peak and hole	1.201 and -1.038 $e.Å^{-3}$	

Table 26.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^{1a})(gly)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	-3601(1)	-3123(1)	-28(1)	33(1)
S(1)	-3663(1)	-1506(1)	201(2)	42(1)
O(10)	-3160(4)	-3394(4)	1379(5)	49(1)
O(14)	-6040(4)	-2576(4)	-2366(5)	54(1)
O(15)	-4526(4)	-2844(3)	-1579(4)	39(1)
O(57)	-3716(4)	-4407(3)	-780(5)	43(1)
N(3)	-1831(4)	-1560(4)	-794(6)	40(1)
N(5)	-2416(4)	-3084(4)	-1078(5)	30(1)
N(6)	-2761(5)	-299(4)	-1272(6)	45(2)
N(11)	-5116(4)	-3350(4)	577(5)	37(1)
C(2)	-2684(5)	-1110(4)	-701(6)	34(1)
C(4)	-1694(5)	-2454(4)	-854(7)	37(2)
C(12)	-5883(7)	-2994(6)	-264(8)	58(2)
C(13)	-5484(5)	-2795(4)	-1530(6)	34(1)
C(31)	-671(5)	-2805(5)	-550(7)	37(2)
C(32)	-591(6)	-3565(5)	175(8)	49(2)
C(33)	350(7)	-3878(6)	559(9)	63(2)
C(34)	1169(8)	-3459(7)	94(11)	75(3)
C(35)	1092(6)	-2733(7)	-635(13)	80(3)
C(36)	150(6)	-2380(5)	-1031(10)	58(2)
C(41)	-3664(6)	298(5)	-1173(8)	45(2)
C(42)	-4463(7)	45(6)	-2082(9)	60(2)
C(43)	-2002(7)	26(5)	-2125(8)	58(2)
C(44)	-1364(10)	732(10)	-1649(13)	100(4)
C(51)	-2248(5)	-3911(4)	-1749(6)	35(2)
C(52)	-1530(6)	-4043(5)	-2623(7)	42(2)
C(53)	-1538(7)	-4861(5)	-3317(8)	52(2)
C(54)	-2228(6)	-5534(5)	-3110(7)	49(2)
C(55)	-2952(6)	-5404(5)	-2254(7)	44(2)
C(56)	-3000(5)	-4592(4)	-1597(6)	38(2)

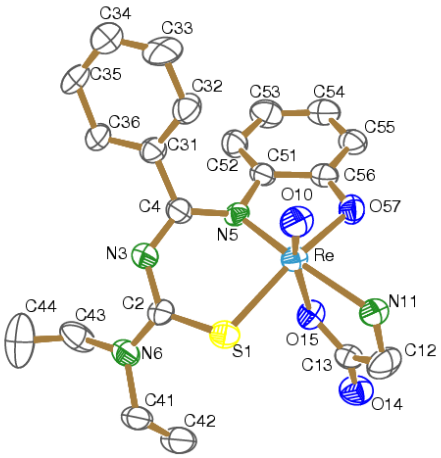

Figure 26 Ellipsoid plot (50% probability) of [ReO(L^{1a})(gly)].

27 [ReO(L^{1a})(Ph_2tbu)], (20a)

Table 27.1 Crystal data and structure refinement for [ReO(L^{1a})(Ph_2tbu)] · EtOH.

Empirical formula	$C_{40}H_{40}N_5O_4ReS_2$	
Formula weight	905.09	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.925(1) Å	α= 81.77(1)°
	b = 12.322(1) Å	β= 85.95(1)°
	c = 15.372(1) Å	γ = 85.26(1)°
Volume	1851.0(3) $Å^3$	
Z	2	
Density (calculated)	1.624 g/cm^3	
Absorption coefficient	3.444 mm^{-1}	
F(000)	908	
Crystal description	Needle	
Crystal color	Red	
Crystal size	0.260 x 0.117 x 0.040 mm^3	
Theta range for data collection	2.68 to 29.26	
Index ranges	-12<=h<=13, -16<=k<=16, -21<=l<=21	
Reflections collected	34500	
Independent reflections	9996 [R(int) = 0.1424]	
Completeness to theta = 29.26°	99.1 %	
Absorption correction	Integration	
Max. and min. transmission	0.8728 and 0.6034	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9996 / 0 / 469	
Goodness-of-fit on F^2	0.963	
Final R indices [I>2sigma(I)]	R1 = 0.0642, wR2 = 0.0926	
R indices (all data)	R1 = 0.1304, wR2 = 0.1087	
Largest diff. peak and hole	1.027 and -2.699 e.$Å^{-3}$	

Table 27.2.Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^{1a})(Ph_2tbu)] · EtOH. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	7056(1)	8916(1)	6691(1)	30(1)
O(10)	6188(5)	9920(4)	6079(3)	34(1)
S(1)	9228(2)	9476(2)	6192(1)	36(1)
C(2)	9812(7)	8397(6)	5615(5)	34(2)
N(3)	9068(6)	8042(5)	5026(4)	35(2)
C(4)	7826(7)	7770(6)	5150(5)	33(2)
N(5)	7120(6)	7693(5)	5951(4)	32(1)
O(57)	5631(5)	8027(4)	7345(3)	36(1)
C(31)	7186(7)	7593(6)	4345(5)	32(2)
C(32)	7945(8)	7141(6)	3669(5)	41(2)
C(33)	7371(9)	7057(7)	2892(6)	52(2)
C(34)	6039(9)	7419(7)	2771(6)	52(2)
C(35)	5279(8)	7870(6)	3433(5)	43(2)
C(36)	5839(8)	7952(6)	4220(5)	40(2)
C(51)	6138(7)	6892(6)	6230(5)	35(2)
C(52)	5972(8)	5960(6)	5869(5)	41(2)
C(53)	5010(9)	5268(7)	6255(6)	46(2)
C(54)	4258(9)	5475(7)	6992(6)	49(2)
C(55)	4443(8)	6395(7)	7378(6)	41(2)
C(56)	5385(7)	7113(6)	6989(5)	36(2)
N(6)	11068(6)	7947(5)	5697(4)	42(2)
C(41)	11978(8)	8251(7)	6334(6)	44(2)
C(42)	12794(8)	9198(8)	5948(6)	53(2)
C(43)	11577(8)	7036(8)	5208(7)	66(3)
C(44)	11135(11)	5934(8)	5648(9)	90(4)
S(11)	7048(2)	9807(2)	7981(1)	40(1)
C(12)	8754(8)	9661(6)	8237(5)	36(2)
N(13)	9490(6)	8701(5)	8364(4)	35(1)
C(14)	9150(7)	7777(6)	8088(5)	35(2)
O(15)	8301(5)	7702(4)	7538(3)	34(1)
C(61)	9914(7)	6748(6)	8455(5)	32(2)
C(62)	9537(8)	5752(6)	8280(5)	41(2)
C(63)	10230(8)	4773(7)	8617(6)	51(2)
C(64)	11338(9)	4790(8)	9111(6)	56(2)

C(65)	11731(9)	5776(8)	9283(7)	58(3)
C(66)	11029(8)	6742(7)	8961(5)	44(2)
N(16)	9328(6)	10552(5)	8399(4)	36(2)
C(71)	8569(7)	11609(6)	8281(6)	40(2)
C(72)	8732(9)	12277(7)	7496(6)	50(2)
C(73)	8016(10)	13305(7)	7337(7)	63(3)
C(74)	7147(11)	13646(9)	7989(9)	75(4)
C(75)	6973(9)	12993(9)	8785(9)	76(4)
C(76)	7688(8)	11951(8)	8953(6)	52(2)
C(81)	10722(7)	10554(6)	8625(6)	41(2)
C(82)	11743(8)	10098(7)	8107(6)	53(2)
C(83)	13089(8)	10082(8)	8339(7)	60(3)
C(84)	13365(9)	10521(7)	9053(6)	55(2)
C(85)	12355(9)	10993(8)	9550(6)	58(2)
C(86)	11009(8)	11004(7)	9348(5)	48(2)
C(87)	4287(11)	4179(11)	507(8)	88(4)
C(88)	4018(18)	3135(17)	644(10)	127(6)
O(89)	5088(9)	2312(10)	652(7)	132(4)

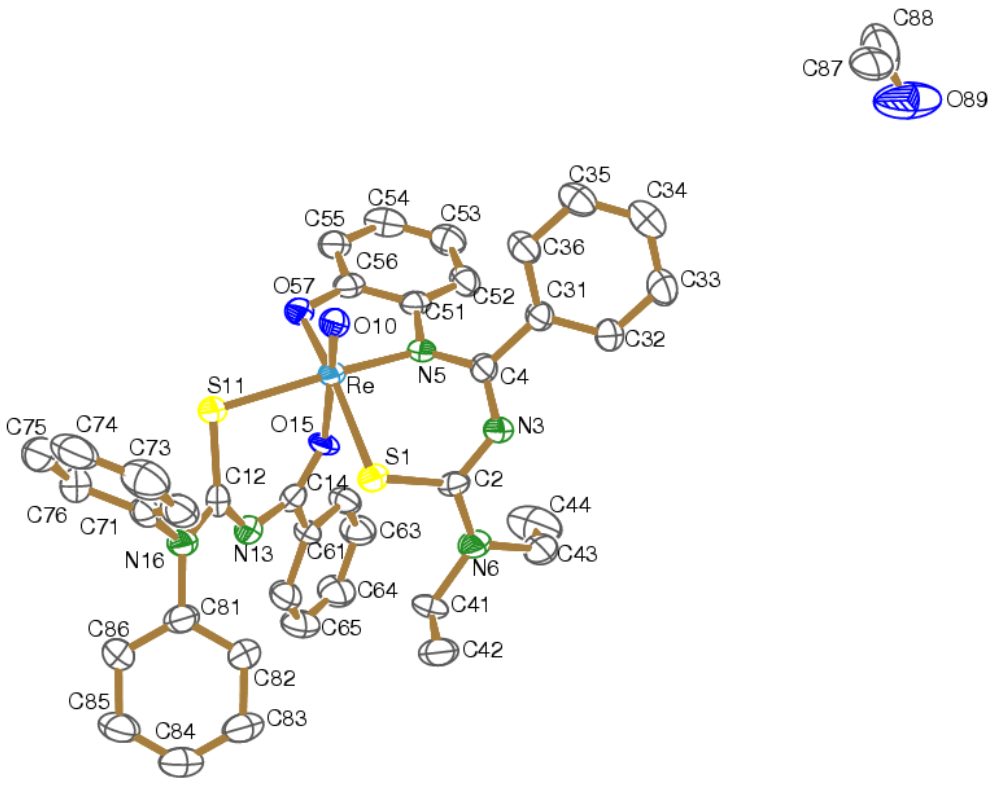

Figure 27 Ellipsoid plot (50% probability) of [ReO(L^{1a})(Ph_2tbu)] · EtOH.

28 [ReO(L^{1a})(morphtbu)], (20b)

Table 28.1 Crystal data and structure refinement for [ReO(L^{1a})(morphtbu)].

Empirical formula	$C_{30}H_{32}N_5O_4ReS_2$	
Formula weight	776.93	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 10.645(1) Å	α= 90.77(1)°
	b = 11.629(1) Å	β= 95.67(1)°
	c = 13.541(1) Å	γ = 113.53(1)°
Volume	1526.9(2) $Å^3$	
Z	2	
Density (calculated)	1.690 g/cm^3	
Absorption coefficient	4.160 mm^{-1}	
F(000)	772	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.9 x 0.8 x 0.55 mm^3	
Theta range for data collection	2.45 to 29.17	
Index ranges	-14<=h<=14, -15<=k<=15, -18<=l<=18	
Reflections collected	27996	
Independent reflections	8144 [R(int) = 0.0974]	
Completeness to theta = 29.17°	98.6 %	
Absorption correction	Integration	
Max. and min. transmission	0.2818 and 0.1311	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8144 / 0 / 379	
Goodness-of-fit on F^2	1.163	
Final R indices [I>2sigma(I)]	R1 = 0.0252, wR2 = 0.0608	
R indices (all data)	R1 = 0.0265, wR2 = 0.0613	
Largest diff. peak and hole	0.944 and -1.509 e.$Å^{-3}$	

Table 28.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^{1a})(morphtbu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	9463(1)	8015(1)	7519(1)	20(1)
O(10)	10943(2)	8019(2)	8084(2)	30(1)
S(1)	9451(1)	9614(1)	8599(1)	24(1)
C(2)	8076(3)	8801(2)	9274(2)	23(1)
N(3)	7736(2)	7617(2)	9544(2)	25(1)
C(4)	7768(2)	6637(2)	9075(2)	22(1)
N(5)	8002(2)	6562(2)	8116(1)	22(1)
C(51)	7402(3)	5448(2)	7480(2)	23(1)
C(56)	7965(3)	5585(2)	6573(2)	24(1)
C(52)	6299(3)	4328(2)	7637(2)	29(1)
C(53)	5821(3)	3349(3)	6914(2)	35(1)
C(54)	6407(3)	3479(3)	6026(2)	36(1)
C(55)	7461(3)	4605(3)	5848(2)	32(1)
O(57)	8972(2)	6708(2)	6412(1)	26(1)
C(31)	7570(3)	5558(2)	9723(2)	22(1)
C(32)	6707(3)	5359(2)	10480(2)	28(1)
C(33)	6568(3)	4394(3)	11116(2)	35(1)
C(34)	7298(3)	3661(3)	11025(2)	36(1)
C(35)	8176(3)	3874(2)	10289(2)	32(1)
C(36)	8296(3)	4811(2)	9631(2)	27(1)
N(6)	7365(2)	9410(2)	9612(2)	26(1)
C(41)	6165(3)	8757(3)	10154(2)	35(1)
C(42)	6580(4)	8815(4)	11259(3)	49(1)
C(43)	7779(3)	10766(2)	9541(2)	29(1)
C(44)	7078(4)	11083(3)	8613(2)	44(1)
S(11)	10394(1)	9529(1)	6304(1)	26(1)
C(12)	9534(3)	10535(2)	6309(2)	23(1)
N(13)	8179(2)	10195(2)	6359(2)	24(1)
C(14)	7319(3)	9036(2)	6538(2)	21(1)
O(15)	7610(2)	8133(2)	6819(1)	25(1)
C(61)	5829(3)	8795(2)	6435(2)	23(1)
C(62)	5370(3)	9736(3)	6214(2)	31(1)
C(63)	3976(3)	9487(3)	6149(2)	37(1)
C(64)	3029(3)	8288(3)	6305(2)	35(1)

C(65)	3475(3)	7350(3)	6525(2)	33(1)
C(66)	4868(3)	7591(2)	6594(2)	28(1)
N(16)	10246(2)	11743(2)	6158(2)	27(1)
C(71)	11712(3)	12319(3)	6025(2)	33(1)
C(72)	12424(3)	13464(3)	6729(3)	42(1)
O(73)	11793(3)	14331(2)	6614(2)	45(1)
C(75)	9604(3)	12643(2)	6043(3)	36(1)
C(74)	10380(4)	13758(3)	6778(3)	44(1)

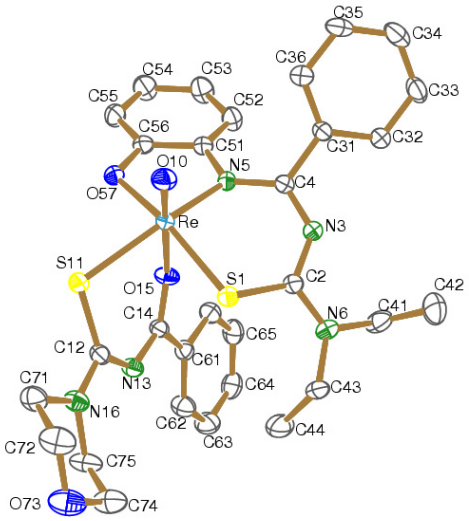

Figure 28 Ellipsoid plot (50% probability) of [ReO(L^{1a})(morphtbu)].

29 [TcO(L^{1b})(morphtbu)], (21b)

Table 29.1 Crystal data and structure refinement for [TcO(L^{1b})(morphtbu)].

Empirical formula	$C_{30}H_{30}N_5O_5S_2Tc$	
Formula weight	703.40	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.565(1) Å	α= 90°
	b = 33.082(3) Å	β= 97.89(1)°
	c = 8.604(1) Å	γ = 90°
Volume	2978.7(5) $Å^3$	
Z	4	
Density (calculated)	1.568 g/cm^3	
Absorption coefficient	0.673 mm^{-1}	
F(000)	1440	
Crystal description	Block	
Crystal color	Green	
Crystal size	0.400 x 0.333 x 0.250 mm^3	
Theta range for data collection	1.95 to 26.85	
Index ranges	-13<=h<=13, -41<=k<=37, -8<=l<=10	
Reflections collected	14209	
Independent reflections	6265 [R(int) = 0.1073]	
Completeness to theta = 26.85°	98.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6265 / 0 / 418	
Goodness-of-fit on F^2	0.909	
Final R indices [I>2sigma(I)]	R1 = 0.0529, wR2 = 0.0991	
R indices (all data)	R1 = 0.1276, wR2 = 0.1362	
Largest diff. peak and hole	0.657 and -0.761 $e.Å^{-3}$	

Table 29.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcO(L^{1b})(morphtbu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	-2821(1)	-1075(1)	-2095(1)	37(1)
S(1)	-3244(2)	-1175(1)	-4814(2)	48(1)
S(11)	-3749(1)	-412(1)	-2559(2)	44(1)
O(57)	-1944(4)	-901(1)	4(5)	41(1)
O(10)	-4084(4)	-1294(1)	-1549(6)	52(1)
O(15)	-1179(3)	-747(1)	-2796(5)	37(1)
O(43)	-4645(4)	-2412(1)	-8405(6)	61(1)
O(73)	-3877(4)	1083(1)	-1089(6)	57(1)
N(3)	-1573(4)	-1801(1)	-4313(6)	39(1)
N(5)	-1458(4)	-1519(1)	-1783(6)	35(1)
N(6)	-3280(6)	-1897(2)	-6183(8)	61(2)
N(13)	-1345(4)	-80(1)	-1927(6)	38(1)
N(16)	-2980(4)	283(1)	-1201(7)	46(1)
C(2)	-2668(6)	-1663(2)	-5051(8)	47(2)
C(4)	-928(5)	-1691(2)	-2972(7)	34(1)
C(12)	-2586(5)	-62(2)	-1796(8)	40(1)
C(14)	-780(5)	-388(2)	-2559(7)	35(1)
C(31)	469(5)	-1773(2)	-2840(7)	37(1)
C(32)	919(6)	-2112(2)	-3545(8)	48(2)
C(33)	2198(7)	-2163(2)	-3581(10)	57(2)
C(34)	3071(7)	-1878(2)	-2952(10)	65(2)
C(35)	2626(6)	-1536(2)	-2272(9)	58(2)
C(36)	1348(5)	-1483(2)	-2214(8)	43(2)
C(41)	-2754(8)	-2290(2)	-6601(12)	73(3)
C(42)	-3753(9)	-2574(2)	-7122(12)	83(3)
C(44)	-5213(8)	-2050(2)	-7912(14)	83(3)
C(45)	-4315(10)	-1748(3)	-7387(15)	98(4)
C(51)	-859(5)	-1524(2)	-190(7)	35(1)
C(52)	-100(6)	-1833(2)	516(8)	41(2)
C(53)	387(7)	-1814(2)	2099(8)	46(2)
C(54)	113(6)	-1479(2)	2951(8)	48(2)
C(55)	-665(6)	-1171(2)	2282(8)	43(2)
C(56)	-1167(5)	-1192(2)	711(8)	37(1)
C(61)	499(5)	-289(2)	-3027(8)	36(1)

C(62)	1064(6)	-543(2)	-3982(9)	51(2)
C(63)	2258(7)	-455(3)	-4382(9)	62(2)
C(64)	2859(7)	-108(3)	-3864(10)	63(2)
C(65)	2328(6)	147(2)	-2871(11)	64(2)
C(66)	1145(6)	57(2)	-2462(10)	52(2)
C(71)	-2100(6)	624(2)	-740(11)	58(2)
C(72)	-2652(6)	1005(2)	-1473(9)	52(2)
C(74)	-4708(6)	759(2)	-1577(12)	65(2)
C(75)	-4259(6)	370(2)	-867(10)	51(2)

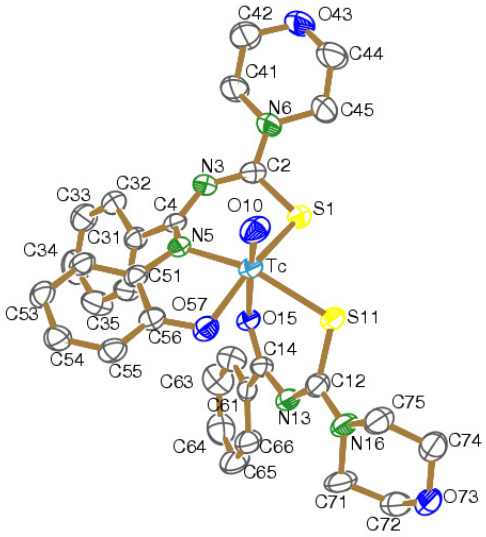

Figure 29 Ellipsoid plot (50% probability) of [TcO(L^{1b})(morphtbu)].

30 [Tc(PPh$_3$)(morphbtu)(L^{1a})], (22b)

Table 30.1 Crystal data and structure refinement for [Tc(PPh$_3$)(morphbtu)(L^{1a})].

Empirical formula	$C_{48}H_{45}N_5O_4PS_2Tc$	
Formula weight	948.98	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 15.973(3) Å	α= 90°
	b = 11.463(1) Å	β= 95.67(1)°
	c = 23.687(5) Å	γ = 90°
Volume	4316.1(1) Å^3	
Z	4	
Density (calculated)	1.460 g/cm^3	
Absorption coefficient	0.519 mm^{-1}	
F(000)	1960	
Crystal description	Needle	
Crystal color	Red	
Crystal size	0.22 x 0.10 x 0.04 mm^3	
Theta range for data collection	2.31 to 26.87	
Index ranges	-20<=h<=20, -14<=k<=14, -29<=l<=29	
Reflections collected	34742	
Independent reflections	9157 [R(int) = 0.4936]	
Completeness to theta = 26.87°	98.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9157 / 0 / 550	
Goodness-of-fit on F^2	0.776	
Final R indices [I>2sigma(I)]	R1 = 0.0873, wR2 = 0.1304	
R indices (all data)	R1 = 0.3190, wR2 = 0.2080	
Largest diff. peak and hole	0.490 and -1.075 e.Å^{-3}	

Table 30.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Tc(PPh_3)(morphbtu)(L^{1a})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	6911(1)	8048(1)	5818(1)	38(1)
S(1)	6808(2)	9708(3)	6386(2)	51(1)
C(2)	7466(9)	9424(11)	7005(7)	51(4)
N(3)	7537(6)	8386(8)	7262(5)	46(3)
C(4)	7445(7)	7303(10)	7019(7)	42(4)
N(5)	7510(6)	7088(9)	6493(4)	35(3)
C(31)	7226(8)	6401(10)	7452(6)	40(3)
C(32)	7558(9)	6454(11)	8007(7)	49(4)
C(33)	7310(11)	5640(14)	8395(7)	71(5)
C(34)	6706(10)	4820(13)	8198(7)	60(5)
C(35)	6337(9)	4813(14)	7683(7)	61(4)
C(36)	6603(8)	5595(11)	7281(6)	45(4)
N(6)	7869(7)	10331(9)	7281(5)	59(4)
C(41)	7809(10)	11525(11)	7090(7)	72(5)
C(42)	7776(11)	12341(11)	7569(7)	72(5)
O(43)	8465(7)	12190(8)	7966(5)	77(3)
C(44)	8481(12)	11033(12)	8217(8)	93(7)
C(45)	8551(8)	10146(12)	7732(7)	66(5)
C(51)	7764(8)	5921(10)	6304(6)	38(3)
C(52)	8286(9)	5176(11)	6626(7)	59(4)
C(53)	8494(9)	4122(11)	6378(7)	53(4)
C(54)	8195(9)	3881(11)	5815(7)	53(4)
C(55)	7716(8)	4637(10)	5494(6)	45(4)
C(56)	7512(8)	5721(11)	5733(7)	50(4)
O(57)	7092(5)	6535(7)	5386(4)	47(2)
S(11)	6083(2)	8777(3)	5025(2)	48(1)
C(12)	5298(8)	7744(10)	4877(7)	47(4)
N(13)	4967(6)	6999(10)	5224(5)	46(3)
C(14)	5206(8)	6890(12)	5778(6)	40(3)
O(15)	5839(5)	7281(7)	6078(4)	52(3)
C(61)	4600(8)	6106(11)	6073(7)	48(4)
C(62)	4581(9)	6222(12)	6634(7)	53(4)
C(63)	4038(9)	5451(15)	6898(8)	77(5)
C(64)	3580(9)	4643(14)	6604(9)	71(5)

C(65)	3604(8)	4540(12)	6031(8)	52(4)
C(66)	4109(8)	5268(11)	5762(7)	49(4)
N(16)	4920(7)	7772(9)	4330(5)	57(3)
C(71)	4122(8)	7123(13)	4160(6)	60(4)
C(72)	4085(10)	6701(13)	3593(6)	66(5)
O(73)	4225(7)	7595(8)	3201(4)	71(3)
C(74)	5035(9)	8083(17)	3327(7)	88(6)
C(75)	5158(10)	8583(13)	3900(7)	73(5)
P	8156(2)	8916(3)	5498(2)	38(1)
C(81)	8969(7)	9378(11)	6036(6)	41(4)
C(82)	9262(7)	8641(12)	6460(6)	47(4)
C(83)	9923(8)	8881(14)	6845(7)	63(5)
C(84)	10290(11)	9920(18)	6840(8)	84(6)
C(85)	10054(9)	10732(14)	6418(8)	70(5)
C(86)	9376(7)	10506(11)	5999(6)	46(4)
C(91)	8703(7)	7880(10)	5074(5)	32(3)
C(92)	9513(8)	7532(11)	5198(7)	54(4)
C(93)	9866(9)	6713(12)	4860(8)	64(5)
C(94)	9437(11)	6274(13)	4380(8)	72(5)
C(95)	8638(10)	6611(12)	4274(8)	67(5)
C(96)	8258(9)	7413(12)	4590(7)	54(4)
C(101)	8005(7)	10195(9)	5027(5)	29(3)
C(102)	8528(9)	10450(11)	4638(6)	50(4)
C(103)	8377(9)	11402(12)	4274(7)	61(5)
C(104)	7692(9)	12100(13)	4335(7)	64(5)
C(105)	7180(8)	11885(13)	4728(6)	53(4)
C(106)	7328(7)	10905(10)	5073(6)	42(4)

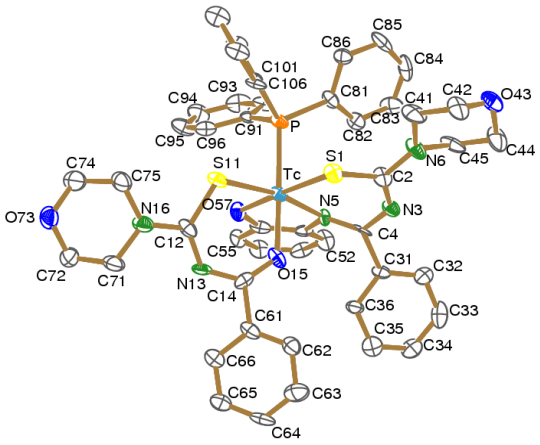

Figure 30 Ellipsoid plot (50% probability) of [Tc(PPh$_3$)(morphbtu)(L^{1a})].

31 [ReNCl(L^{1b})(PPh_3)], (23b)

Table 31.1 Crystal data and structure refinement for [ReNCl(L^{1b})(PPh_3)].

Empirical formula	$C_{36}H_{32}N_4O_2PReS$	
Formula weight	801.89	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.473(1) Å	α= 95.04(1)°
	b = 11.650(1) Å	β= 95.96(1)°
	c = 15.423(1) Å	γ = 108.32(1)°
Volume	1593.9(2) $Å^3$	
Z	2	
Density (calculated)	1.671 g/cm^3	
Absorption coefficient	3.968 mm^{-1}	
F(000)	796	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.430 x 0.303 x 0.240 mm^3	
Theta range for data collection	1.86 to 29.31	
Index ranges	-11<=h<=12, -15<=k<=14, -21<=l<=21	
Reflections collected	17391	
Independent reflections	8499 [R(int) = 0.1023]	
Completeness to theta = 29.31°	97.5 %	
Absorption correction	Integration	
Max. and min. transmission	0.4805 and 0.2936	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8499 / 0 / 406	
Goodness-of-fit on F^2	0.937	
Final R indices [I>2sigma(I)]	R1 = 0.0422, wR2 = 0.0901	
R indices (all data)	R1 = 0.0649, wR2 = 0.1094	
Largest diff. peak and hole	1.791 and -3.067 e.$Å^{-3}$	

Table 31.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReNCl(L^{1b})(PPh_3)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	8420(1)	8565(1)	7439(1)	28(1)
N(10)	9996(6)	9646(6)	7364(4)	42(1)
S(1)	8913(2)	7776(2)	8720(1)	36(1)
C(2)	8253(7)	8533(6)	9547(4)	34(1)
N(3)	8078(6)	9625(5)	9517(3)	32(1)
C(4)	7617(6)	10056(5)	8825(4)	29(1)
N(5)	7034(5)	9376(5)	8035(3)	33(1)
C(31)	7804(6)	11382(5)	8959(4)	32(1)
C(32)	8026(7)	12086(6)	8260(5)	40(1)
C(33)	8303(8)	13330(6)	8396(5)	46(2)
C(34)	8344(9)	13884(7)	9233(6)	52(2)
C(35)	8125(8)	13223(7)	9920(5)	46(2)
C(36)	7856(7)	11966(6)	9796(4)	38(1)
N(6)	7981(6)	8010(5)	10277(4)	37(1)
C(41)	8269(8)	6897(6)	10473(5)	43(2)
C(42)	6829(9)	5950(7)	10538(6)	61(2)
O(43)	6054(7)	6337(5)	11191(4)	65(2)
C(44)	5790(9)	7408(8)	11005(6)	60(2)
C(45)	7192(9)	8422(7)	10950(4)	48(2)
C(51)	5933(6)	9596(6)	7432(4)	32(1)
C(52)	4887(7)	10153(6)	7647(4)	40(2)
C(53)	3861(7)	10277(7)	6980(5)	43(2)
C(54)	3832(8)	9804(7)	6133(5)	45(2)
C(55)	4808(7)	9181(7)	5936(4)	41(2)
C(56)	5860(6)	9067(5)	6572(4)	30(1)
O(57)	6805(5)	8440(4)	6431(3)	36(1)
P	9095(2)	7054(1)	6552(1)	28(1)
C(61)	7519(6)	5648(5)	6241(4)	33(1)
C(62)	6685(8)	5148(7)	6885(5)	46(2)
C(63)	5513(9)	4054(8)	6680(7)	62(2)
C(64)	5152(8)	3480(7)	5828(6)	56(2)
C(65)	5948(8)	3974(7)	5179(5)	48(2)
C(66)	7126(7)	5050(6)	5381(4)	40(1)
C(71)	9698(6)	7542(5)	5519(4)	28(1)

C(72)	10852(7)	7257(6)	5159(4)	36(1)
C(73)	11282(8)	7663(7)	4382(4)	43(2)
C(74)	10552(8)	8334(7)	3959(4)	45(2)
C(75)	9385(8)	8619(7)	4294(5)	47(2)
C(76)	8974(7)	8230(7)	5078(4)	39(1)
C(81)	10612(7)	6544(6)	7032(4)	32(1)
C(82)	10623(8)	5360(7)	6832(5)	43(2)
C(83)	11842(9)	5046(8)	7131(6)	56(2)
C(84)	13059(9)	5883(9)	7638(5)	56(2)
C(85)	13072(8)	7067(8)	7855(5)	51(2)
C(86)	11835(7)	7401(7)	7554(4)	40(1)

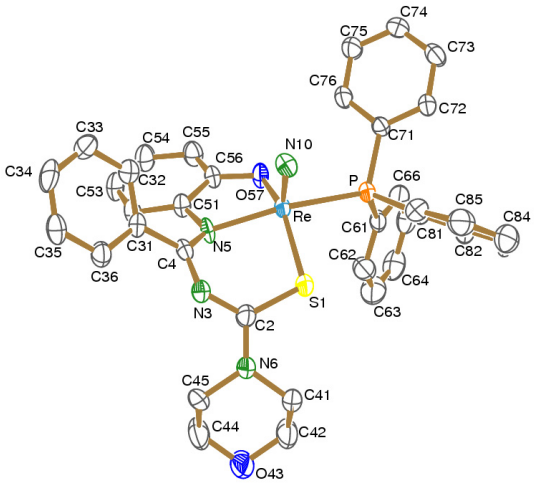

Figure 31 Ellipsoid plot (50% probability) of [ReNCl(L^{1b})(PPh_3)].

32 [TcNCl(L^{1b})(PPh_3)], (24b)

Table 32.1 Crystal data and structure refinement for [TcNCl(L^{1b})(PPh_3)].

Empirical formula	$C_{36}H_{32}N_4O_2PSTc$	
Formula weight	713.69	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.454(1) Å	α= 94.76(1)°
	b = 11.656(1) Å	β= 95.41(1)°
	c = 15.451(1) Å	γ = 108.45(1)°
Volume	1596.3(2) $Å^3$	
Z	2	
Density (calculated)	1.485 g/cm^3	
Absorption coefficient	0.606 mm^{-1}	
F(000)	732	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.20 x 0.15 x 0.10 mm^3	
Theta range for data collection	1.86 to 29.31	
Index ranges	-11<=h<=12, -15<=k<=14, -21<=l<=21	
Reflections collected	17115	
Independent reflections	8518 [R(int) = 0.1245]	
Completeness to theta = 29.31°	97.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8518 / 0 / 407	
Goodness-of-fit on F^2	0.988	
Final R indices [I>2sigma(I)]	R1 = 0.0711, wR2 = 0.1378	
R indices (all data)	R1 = 0.1339, wR2 = 0.1756	
Extinction coefficient	0.036(2)	
Largest diff. peak and hole	0.945 and -0.754 e.$Å^{-3}$	

Table 32.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcNCl(L^{1b})(PPh_3)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	8417(1)	8548(1)	7430(1)	26(1)
N(10)	9970(6)	9617(5)	7351(3)	34(1)
S(1)	8899(2)	7738(1)	8710(1)	36(1)
C(2)	8279(7)	8506(5)	9544(4)	30(1)
N(3)	8110(6)	9603(4)	9519(3)	31(1)
C(4)	7619(7)	10028(5)	8815(4)	29(1)
N(5)	7035(6)	9352(4)	8053(3)	31(1)
C(31)	7788(7)	11364(5)	8967(4)	29(1)
C(32)	7997(8)	12058(6)	8270(4)	36(1)
C(33)	8263(8)	13297(6)	8399(5)	42(2)
C(34)	8299(9)	13845(6)	9235(5)	47(2)
C(35)	8074(8)	13160(6)	9927(5)	43(2)
C(36)	7824(7)	11931(5)	9797(4)	33(1)
N(6)	8046(7)	8008(5)	10284(3)	37(1)
C(41)	7285(9)	8438(6)	10968(5)	45(2)
C(42)	5841(9)	7445(7)	11007(6)	52(2)
O(43)	6052(7)	6339(5)	11187(4)	60(2)
C(44)	6801(10)	5946(6)	10525(6)	54(2)
C(45)	8293(8)	6876(6)	10468(5)	40(2)
C(51)	5937(7)	9574(5)	7451(4)	31(1)
C(52)	4886(7)	10127(6)	7668(4)	37(1)
C(53)	3873(8)	10262(7)	7013(5)	44(2)
C(54)	3851(8)	9806(6)	6166(5)	41(2)
C(55)	4829(8)	9190(6)	5950(5)	39(2)
C(56)	5867(7)	9045(5)	6594(4)	30(1)
O(57)	6795(5)	8406(4)	6430(3)	33(1)
P	9084(2)	7017(1)	6543(1)	27(1)
C(61)	9690(7)	7522(5)	5511(4)	28(1)
C(62)	10895(8)	7276(6)	5165(4)	36(1)
C(63)	11336(8)	7717(6)	4392(4)	38(2)
C(64)	10613(9)	8393(6)	3970(4)	46(2)
C(65)	9405(9)	8623(7)	4295(5)	46(2)
C(66)	8971(8)	8202(6)	5072(4)	38(2)
C(71)	10586(7)	6499(6)	7008(4)	32(1)

C(72)	11798(8)	7330(7)	7519(5)	42(2)
C(73)	13037(9)	7000(7)	7815(5)	48(2)
C(74)	13023(9)	5814(8)	7615(5)	53(2)
C(75)	11805(10)	4985(7)	7117(6)	54(2)
C(76)	10583(8)	5312(6)	6811(5)	41(2)
C(81)	7488(7)	5616(5)	6221(4)	30(1)
C(82)	7137(8)	5052(6)	5378(5)	39(1)
C(83)	5916(9)	3956(6)	5175(5)	45(2)
C(84)	5120(9)	3460(7)	5823(6)	53(2)
C(85)	5475(9)	4025(7)	6666(6)	55(2)
C(86)	6647(8)	5105(6)	6864(5)	43(2)

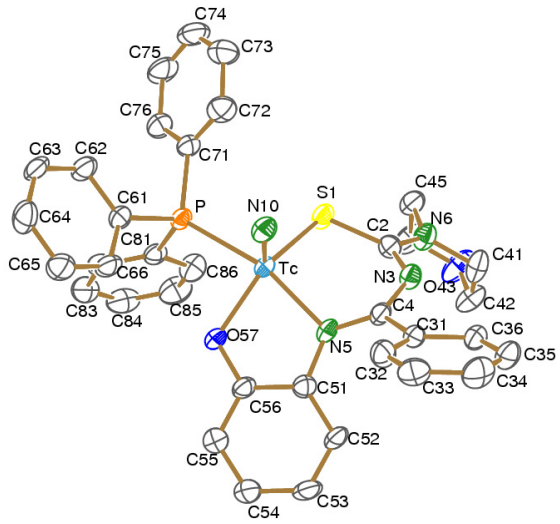

Figure 32 Ellipsoid plot (50% probability) of [TcNCl(L^{1b})(PPh_3)].

33 [{ReOCl(L^2)}$_2$O], (25)

Table 33.1 Crystal data and structure refinement for [{ReOCl(L^2)}$_2$O].

Empirical formula	$C_{36}H_{42}ClN_4O_3Re_2S_2$	
Formula weight	1142.20	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 10.003(1) Å	α= 90°
	b = 10.630(1) Å	β= 99.54(1)°
	c = 19.441(2) Å	γ = 90°
Volume	2038.5(3) Å^3	
Z	4	
Density (calculated)	1.861 g/cm^3	
Absorption coefficient	6.212 mm^{-1}	
F(000)	1108	
Crystal description	Polyhedron	
Crystal color	Violet	
Crystal size	0.400 x 0.197 x 0.040 mm^3	
Theta range for data collection	2.82 to 26.79	
Index ranges	-12<=h<=12, -11<=k<=13, -21<=l<=24	
Reflections collected	7811	
Independent reflections	4181 [R(int) = 0.0551]	
Completeness to theta = 26.79°	95.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.6763 and 0.4677	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4181 / 0 / 241	
Goodness-of-fit on F^2	0.947	
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0650	
R indices (all data)	R1 = 0.0569, wR2 = 0.0704	
Largest diff. peak and hole	1.148 and -1.319 e.Å^{-3}	

Table 33.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReOCl(L^2)}$_2$O]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	6267(1)	1362(1)	167(1)	24(1)
O(2O)	5000	0	0	24(1)
O(10)	7598(5)	2367(4)	261(2)	32(1)
Cl	5881(2)	1657(2)	-1104(1)	38(1)
S(1)	4571(2)	2819(2)	275(1)	29(1)
C(2)	4645(7)	3058(6)	1169(3)	30(1)
N(3)	5254(6)	2378(5)	1700(3)	37(1)
C(4)	6063(6)	1376(6)	1709(3)	25(1)
N(5)	6565(5)	839(5)	1190(3)	29(1)
C(6)	7403(8)	-306(7)	1365(4)	40(2)
C(51)	8083(7)	-704(6)	767(4)	34(2)
N(52)	7680(5)	-162(5)	142(3)	28(1)
C(53)	8235(7)	-533(6)	-411(4)	33(1)
C(54)	9229(8)	-1446(7)	-346(4)	49(2)
C(55)	9636(8)	-1988(7)	291(5)	48(2)
C(56)	9056(7)	-1642(6)	849(4)	40(2)
C(31)	6502(6)	863(6)	2431(3)	28(1)
C(32)	5845(8)	-151(7)	2677(4)	41(2)
C(33)	6339(9)	-604(7)	3343(4)	45(2)
C(34)	7446(8)	-69(7)	3763(4)	43(2)
C(35)	8065(8)	939(7)	3511(4)	43(2)
C(36)	7606(7)	1427(7)	2860(3)	36(1)
N(6)	3960(7)	4065(6)	1355(3)	47(2)
C(41)	3935(12)	4303(10)	2104(4)	78(4)
C(42)	2966(13)	3450(16)	2379(6)	124(6)
C(43)	3309(8)	5004(7)	859(4)	43(2)
C(44)	1829(10)	4828(9)	652(6)	73(3)

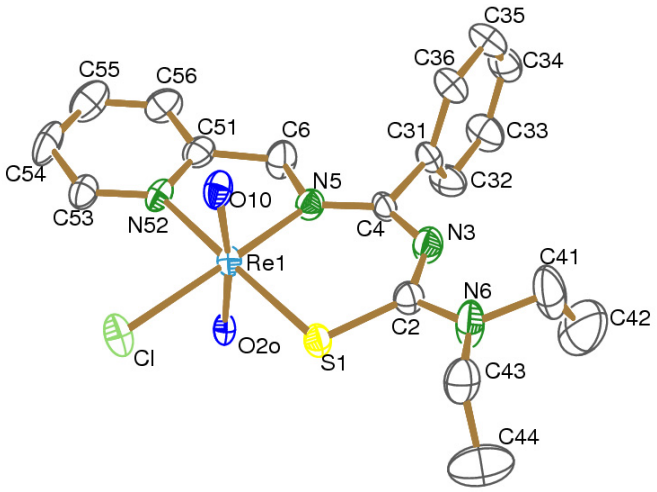

Figure 33 Ellipsoid plot (50% probability) of the asymmetric unit of [{ReOCl(L^2)}$_2$O].

34 [{ReOCl(L^3)}$_2$], (26)

Table 34.1 Crystal data and structure refinement for [{ReOCl(L^3)}$_2$].

Empirical formula	$C_{38}H_{38}Cl_2N_6O_6Re_2S_2$	
Formula weight	1182.16	
Temperature	290(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 8.865(1) Å	α= 90°
	b = 13.704(1) Å	β= 90.91(1)°
	c = 16.230(1) Å	γ = 90°
Volume	1971.4(3) Å^3	
Z	2	
Density (calculated)	1.991 g/cm^3	
Absorption coefficient	6.431 mm^{-1}	
F(000)	1144	
Crystal description	Needle	
Crystal color	Green	
Crystal size	0.2 x 0.1 x 0.05 mm^3	
Theta range for data collection	1.95 to 29.24	
Index ranges	-12<=h<=11, -18<=k<=18, -22<=l<=18	
Reflections collected	14571	
Independent reflections	5306 [R(int) = 0.1396]	
Completeness to theta = 29.24°	98.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.9118 and 0.7214	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5306 / 0 / 253	
Goodness-of-fit on F^2	0.914	
Final R indices [I>2sigma(I)]	R1 = 0.0532, wR2 = 0.0940	
R indices (all data)	R1 = 0.1245, wR2 = 0.1340	
Largest diff. peak and hole	2.200 and -2.205 e.Å^{-3}	

Table 34.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReOCl(L^3)}$_2$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	846(1)	4828(1)	1575(1)	20(1)
O(10)	-203(9)	4814(6)	2409(5)	32(2)
Cl	1476(3)	6512(2)	1636(2)	30(1)
S(1)	3207(3)	4545(2)	2124(2)	26(1)
C(2)	3354(12)	3270(8)	2184(7)	24(2)
N(6)	4240(11)	2889(7)	2771(6)	27(2)
C(43)	5103(14)	3496(9)	3359(7)	32(3)
C(44)	6607(14)	3812(9)	3033(9)	38(3)
C(41)	4478(15)	1822(8)	2832(8)	30(3)
C(42)	3356(17)	1350(9)	3397(10)	43(4)
N(3)	2757(10)	2654(6)	1631(6)	23(2)
C(4)	1532(13)	2730(7)	1150(7)	23(2)
C(31)	1452(12)	1972(7)	492(7)	21(2)
C(36)	2020(17)	1049(8)	636(8)	39(3)
C(35)	2040(16)	343(8)	31(7)	35(3)
C(34)	1447(17)	592(8)	-726(8)	38(3)
C(33)	887(13)	1498(9)	-891(7)	29(3)
C(32)	879(13)	2200(8)	-279(7)	22(2)
N(5)	466(11)	3421(6)	1217(6)	22(2)
C(51)	-1064(12)	3158(8)	999(6)	20(2)
C(52)	-1613(16)	2235(9)	1261(8)	33(3)
C(53)	-3073(14)	1949(9)	1064(8)	31(3)
C(54)	-4007(14)	2534(9)	616(8)	31(3)
C(55)	-3520(13)	3430(9)	363(8)	33(3)
C(56)	-2046(11)	3756(8)	558(6)	19(2)
C(57)	-1596(10)	4739(8)	261(6)	20(2)
O(57)	-710(7)	5267(6)	702(4)	23(2)
O(58)	-2164(8)	5074(5)	-389(4)	23(2)

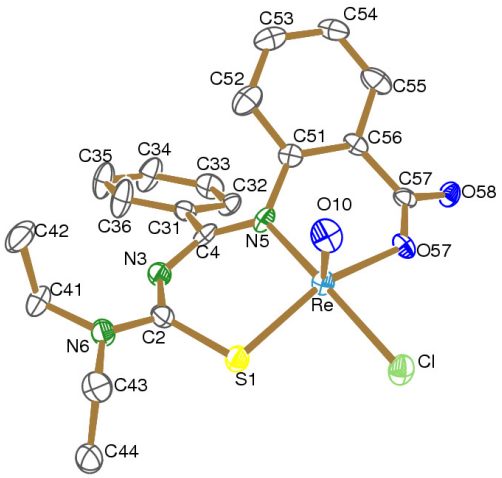

Figure 34 Ellipsoid plot (50% probability) of [{ReOCl(L^3)}$_2$].

35 [ReOCl(L^4)], (27)

Table 35.1 Crystal data and structure refinement for [ReOCl(L^4)].

Empirical formula	$C_{19}H_{20}ClN_4O_2ReS$	
Formula weight	590.10	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.718(1) Å	α= 90°
	b = 13.510(1) Å	β= 95.28(1)°
	c = 15.564(1) Å	γ = 90°
Volume	2034.7(3) $Å^3$	
Z	4	
Density (calculated)	1.926 g/cm^3	
Absorption coefficient	6.229 mm^{-1}	
F(000)	1144	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.200 x 0.167 x 0.100 mm^3	
Theta range for data collection	2.00 to 29.26	
Index ranges	-10<=h<=13, -18<=k<=18, -21<=l<=21	
Reflections collected	15294	
Independent reflections	5483 [R(int) = 0.0941]	
Completeness to theta = 29.26°	98.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.5528 and 0.1599	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5483 / 0 / 254	
Goodness-of-fit on F^2	0.893	
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.0797	
R indices (all data)	R1 = 0.0911, wR2 = 0.0933	
Extinction coefficient	0.0031(2)	
Largest diff. peak and hole	1.031 and -1.514 $e.Å^{-3}$	

Table 35.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReOCl(L^4)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	5479(1)	1953(1)	7877(1)	47(1)
Cl	5733(2)	643(2)	6915(1)	62(1)
O(10)	4517(5)	2824(4)	7391(4)	64(1)
S(1)	7720(2)	2292(2)	7667(1)	56(1)
C(2)	8456(6)	2923(5)	8579(4)	47(2)
N(3)	7801(5)	3312(4)	9226(3)	44(1)
C(4)	6614(5)	3022(5)	9483(4)	39(1)
N(5)	5803(5)	2290(4)	9116(3)	44(1)
N(59)	4918(5)	1833(4)	9680(3)	45(1)
C(58)	4230(5)	1145(5)	9264(4)	44(2)
O(57)	4393(4)	964(3)	8433(3)	48(1)
N(6)	9836(5)	3034(4)	8646(4)	50(1)
C(41)	10724(6)	2565(6)	8030(5)	57(2)
C(42)	10912(9)	3219(7)	7281(6)	77(2)
C(43)	10565(6)	3559(5)	9381(5)	52(2)
C(44)	11087(8)	2871(6)	10081(5)	65(2)
C(31)	6189(6)	3569(5)	10256(4)	42(1)
C(32)	4800(6)	3803(5)	10337(5)	49(2)
C(33)	4463(7)	4343(5)	11033(5)	54(2)
C(34)	5480(8)	4657(5)	11650(5)	62(2)
C(35)	6817(8)	4428(6)	11562(5)	60(2)
C(36)	7177(7)	3892(5)	10860(5)	52(2)
C(51)	3252(6)	513(5)	9679(4)	46(2)
C(52)	2483(7)	-190(5)	9202(5)	54(2)
C(53)	1548(7)	-779(6)	9588(5)	60(2)
C(54)	1368(7)	-660(6)	10444(6)	62(2)
C(55)	2142(7)	27(6)	10923(5)	62(2)
C(56)	3079(7)	620(5)	10554(5)	56(2)

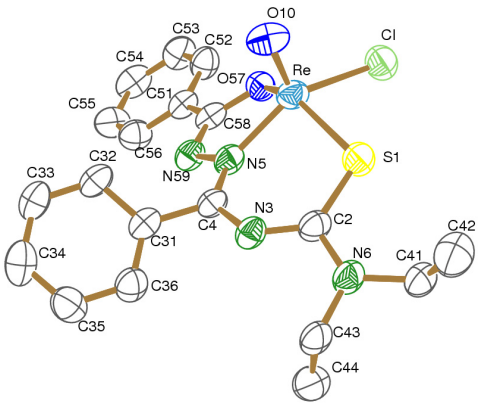

Figure 35 Ellipsoid plot (50% probability) of [ReOCl(L^4)].

36 [TcOCl(L^4)], (28)

Table 36.1 Crystal data and structure refinement for [TcOCl(L^4)].

Empirical formula	$C_{19}H_{20}ClN_4O_2STc$	
Formula weight	501.90	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.148(1) Å	α= 90°
	b = 12.886(1) Å	β= 98.02(1)°
	c = 13.126(1) Å	γ = 90°
Volume	2034.6(3) $Å^3$	
Z	4	
Density (calculated)	1.639 g/cm^3	
Absorption coefficient	0.964 mm^{-1}	
F(000)	1016	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.280 x 0.260 x 0.230 mm^3	
Theta range for data collection	2.14 to 29.27	
Index ranges	-15<=h<=16, -17<=k<=17, -17<=l<=17	
Reflections collected	15331	
Independent reflections	5473 [R(int) = 0.0545]	
Completeness to theta = 29.27°	98.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5473 / 0 / 254	
Goodness-of-fit on F^2	0.948	
Final R indices [I>2sigma(I)]	R1 = 0.0288, wR2 = 0.0618	
R indices (all data)	R1 = 0.0438, wR2 = 0.0658	
Extinction coefficient	0.0068(5)	
Largest diff. peak and hole	0.407 and -0.987 e.$Å^{-3}$	

Table 36.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcOCl(L^4)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	764(1)	4563(1)	1841(1)	26(1)
O(10)	814(1)	4057(1)	2996(1)	37(1)
Cl	-168(1)	6149(1)	1828(1)	39(1)
S(1)	-850(1)	4041(1)	864(1)	32(1)
C(2)	-627(2)	2757(2)	494(2)	28(1)
N(3)	341(1)	2301(1)	400(1)	29(1)
C(4)	1339(2)	2721(2)	559(1)	27(1)
N(5)	1629(1)	3628(1)	1038(1)	28(1)
N(59)	2740(1)	3929(1)	1011(1)	32(1)
C(58)	2912(2)	4839(2)	1395(2)	28(1)
O(57)	2093(1)	5400(1)	1708(1)	30(1)
C(31)	2202(2)	2074(2)	164(2)	29(1)
C(32)	3186(2)	1799(2)	779(2)	39(1)
C(33)	3946(2)	1172(2)	385(2)	47(1)
C(34)	3746(2)	833(2)	-617(2)	49(1)
C(35)	2769(2)	1088(2)	-1231(2)	45(1)
C(36)	1985(2)	1700(2)	-839(2)	35(1)
N(6)	-1518(1)	2154(1)	263(1)	32(1)
C(41)	-2666(2)	2520(2)	239(2)	36(1)
C(42)	-3112(2)	2300(2)	1234(2)	47(1)
C(43)	-1389(2)	1047(2)	-5(2)	45(1)
C(44)	-1312(3)	896(3)	-1130(3)	66(1)
C(51)	4036(2)	5291(2)	1502(2)	30(1)
C(52)	4910(2)	4697(2)	1236(2)	40(1)
C(53)	5969(2)	5111(2)	1311(2)	44(1)
C(54)	6163(2)	6120(2)	1643(2)	43(1)
C(55)	5305(2)	6703(2)	1932(2)	43(1)
C(56)	4247(2)	6292(2)	1865(2)	36(1)

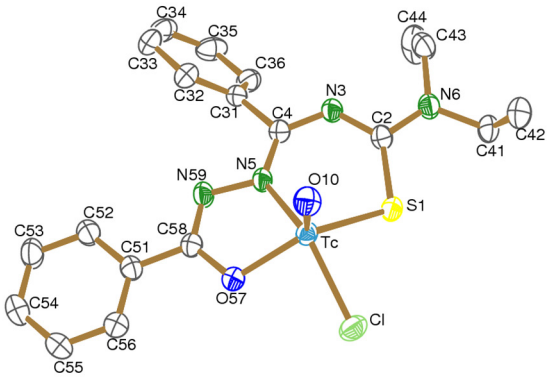

Figure 36 Ellipsoid plot (50% probability) of [TcOCl(L^4)].

37 [ReO(L^4)(Ph_2btu)], (29a)

Table 37.1 Crystal data and structure refinement for [ReO(L^4)(Ph_2btu)].

Empirical formula	$C_{39}H_{35}N_6O_3ReS_2$	
Formula weight	886.05	
Temperature	200(2) K	
Wavelength	0.71069 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 15.940(5) Å	α= 90°
	b = 13.429(5) Å	β= 111.56(1)°
	c = 18.565(5) Å	γ = 90°
Volume	3696(2) $Å^3$	
Z	4	
Density (calculated)	1.592 g/cm^3	
Absorption coefficient	3.447 mm^{-1}	
F(000)	1768	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.5 x 0.3 x 0.2 mm^3	
Theta range for data collection	2.36 to 26.82	
Index ranges	-20<=h<=20, -16<=k<=16, -23<=l<=23	
Reflections collected	12885	
Independent reflections	7180 [R(int) = 0.0669]	
Completeness to theta = 26.82°	98.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.6309 and 0.4792	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7180 / 2 / 461	
Goodness-of-fit on F^2	0.953	
Final R indices [I>2sigma(I)]	R1 = 0.0415, wR2 = 0.0884	
R indices (all data)	R1 = 0.0548, wR2 = 0.0922	
Absolute structure parameter	-0.006(9)	
Extinction coefficient	0.00260(16)	
Largest diff. peak and hole	1.577 and -2.414 e.$Å^{-3}$	

Table 37.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^4)(Ph_2btu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	4966(1)	6573(1)	6582(1)	32(1)
O(10)	5019(5)	6404(5)	7484(3)	46(2)
S(1)	5904(2)	7924(2)	6687(1)	40(1)
C(2)	5221(6)	8917(6)	6717(4)	31(2)
N(3)	4319(6)	9022(6)	6266(4)	40(2)
C(4)	3738(5)	8327(6)	5912(4)	35(2)
N(5)	3809(5)	7372(5)	6108(3)	36(2)
N(59)	3010(5)	6785(5)	5765(3)	36(2)
C(58)	3210(5)	5847(6)	5816(4)	32(2)
O(57)	4039(4)	5480(4)	6107(3)	36(1)
C(31)	2956(6)	8720(6)	5244(5)	41(2)
C(32)	2653(6)	8220(6)	4537(5)	43(2)
C(33)	2017(7)	8662(7)	3886(6)	54(2)
C(34)	1667(7)	9577(8)	3961(5)	60(3)
C(35)	1926(6)	10061(7)	4658(6)	53(2)
C(36)	2586(6)	9627(7)	5306(5)	47(2)
N(6)	5571(5)	9698(6)	7204(4)	49(2)
C(41)	5001(8)	10454(7)	7287(6)	71(4)
C(42)	4459(11)	10148(11)	7804(7)	92(5)
C(43)	6537(8)	9749(9)	7649(5)	64(3)
C(44)	6774(9)	9415(14)	8483(6)	96(5)
C(51)	2475(6)	5096(6)	5525(4)	39(2)
C(52)	1599(6)	5300(7)	5426(5)	47(2)
C(53)	950(7)	4561(9)	5154(6)	62(3)
C(54)	1165(8)	3621(8)	4983(6)	58(3)
C(55)	2048(7)	3419(8)	5062(5)	52(2)
C(56)	2701(7)	4146(7)	5341(4)	44(2)
S(11)	6178(1)	5544(2)	6552(1)	42(1)
C(12)	5538(5)	4689(7)	5838(4)	39(2)
N(13)	5007(5)	4947(5)	5114(4)	42(2)
C(14)	4770(6)	5865(6)	4898(4)	34(2)
O(15)	4785(4)	6605(4)	5320(3)	36(1)
C(61)	4369(5)	6045(7)	4031(4)	36(2)
C(62)	4243(6)	5243(8)	3526(5)	50(2)

C(63)	3829(7)	5430(9)	2726(5)	59(3)
C(64)	3526(7)	6346(8)	2452(5)	55(3)
C(65)	3637(7)	7139(8)	2951(5)	53(2)
C(66)	4085(6)	6982(8)	3756(5)	47(2)
N(16)	5634(5)	3711(5)	5997(4)	39(2)
C(71)	5182(6)	2970(7)	5417(5)	43(2)
C(72)	5358(7)	2882(7)	4748(5)	51(2)
C(73)	4904(8)	2159(9)	4205(6)	61(3)
C(74)	4304(8)	1550(8)	4329(6)	64(3)
C(75)	4122(8)	1625(8)	5012(7)	67(3)
C(76)	4579(7)	2346(7)	5563(5)	54(2)
C(81)	6187(6)	3320(6)	6750(5)	40(2)
C(82)	6981(7)	2855(7)	6834(5)	52(2)
C(83)	7499(8)	2433(8)	7551(6)	63(3)
C(84)	7194(8)	2494(8)	8157(6)	63(3)
C(85)	6387(8)	2948(8)	8056(6)	60(3)
C(86)	5870(7)	3395(7)	7355(5)	49(2)

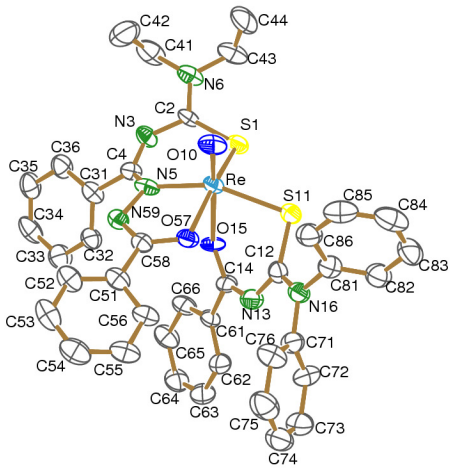

Figure 37 Ellipsoid plot (50% probability) of [ReO(L^4)(Ph_2btu)].

38 [ReOCl(L^{5b})], (30b)

Table 38.1 Crystal data and structure refinement for [ReOCl(L^{5b})].

Empirical formula	$C_{17}H_{23}ClN_5OReS_2$	
Formula weight	599.02	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.982(1) Å	α= 90°
	b = 21.900(1) Å	β= 113.76(1)°
	c = 9.524(1) Å	γ = 90°
Volume	2096.5(3) $Å^3$	
Z	4	
Density (calculated)	1.898 g/cm^3	
Absorption coefficient	6.140 mm^{-1}	
F(000)	1168	
Crystal description	Plate	
Crystal color	Orange	
Crystal size	0.760 x 0.310 x 0.010 mm^3	
Theta range for data collection	1.86 to 29.30	
Index ranges	-15<=h<=15, -30<=k<=29, -13<=l<=12	
Reflections collected	15000	
Independent reflections	5624 [R(int) = 0.0987]	
Completeness to theta = 29.30°	98.1 %	
Absorption correction	Integration	
Max. and min. transmission	0.8484 and 0.2861	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5624 / 0 / 246	
Goodness-of-fit on F^2	0.970	
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1165	
R indices (all data)	R1 = 0.0904, wR2 = 0.1437	
Extinction coefficient	0.0146(7)	
Largest diff. peak and hole	2.220 and -1.468 e.$Å^{-3}$	

Table 38.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReOCl(L^{5b})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	-6803(1)	-849(1)	-6637(1)	38(1)
Cl	-8630(2)	-491(1)	-8814(3)	53(1)
S(1)	-8143(2)	-1701(1)	-7113(3)	52(1)
S(9)	-5556(2)	-403(1)	-7779(3)	42(1)
O(10)	-6819(7)	-449(3)	-5173(8)	54(2)
N(3)	-5978(7)	-2223(3)	-4843(9)	42(2)
N(5)	-5192(6)	-1383(3)	-5801(8)	36(1)
N(6)	-7555(8)	-2869(4)	-6408(10)	54(2)
N(7)	-3942(6)	-1232(3)	-5846(8)	39(2)
N(51)	-2960(7)	-617(4)	-7012(9)	46(2)
C(2)	-7134(9)	-2309(4)	-6071(11)	48(2)
C(4)	-5083(8)	-1799(4)	-4694(9)	36(2)
C(8)	-4032(8)	-793(4)	-6758(10)	41(2)
C(31)	-3918(8)	-1797(4)	-3199(10)	38(2)
C(32)	-3270(9)	-1257(4)	-2566(11)	47(2)
C(33)	-2278(10)	-1253(5)	-1095(12)	56(2)
C(34)	-1935(10)	-1789(5)	-248(12)	61(3)
C(35)	-2581(10)	-2328(5)	-890(12)	56(2)
C(36)	-3550(8)	-2335(4)	-2359(11)	46(2)
C(41)	-6752(10)	-3380(4)	-5492(12)	53(2)
C(42)	-5675(12)	-3558(5)	-6038(15)	66(3)
C(43)	-8808(11)	-3039(5)	-7715(14)	69(3)
C(44)	-9895(12)	-3116(8)	-7208(18)	90(4)
C(52)	-1639(8)	-873(5)	-6107(13)	52(2)
C(53)	-762(10)	-577(5)	-6807(13)	57(2)
C(54)	-1427(11)	44(5)	-7375(12)	58(3)
C(55)	-2913(10)	-97(5)	-7963(11)	52(2)

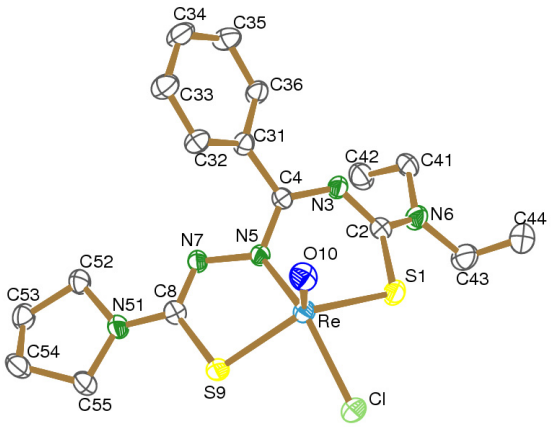

Figure 38 Ellipsoid plot (50% probability) of [ReOCl(L^{5b})].

39 [TcOCl(L^{5a})], (31a)

Table 39.1 Crystal data and structure refinement for [TcOCl(L^{5a})].

Empirical formula	$C_{15}H_{21}ClN_5OS_2Tc$	
Formula weight	485.87	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.012(1) Å	α= 90°
	b = 21.742(2) Å	β= 109.41(1)°
	c = 9.601(1) Å	γ = 90°
Volume	1971.1(3) $Å^3$	
Z	4	
Density (calculated)	1.637 g/cm^3	
Absorption coefficient	1.091 mm^{-1}	
F(000)	984	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.3 x 0.2 x 0.2 mm^3	
Theta range for data collection	2.71 to 29.30	
Index ranges	-13<=h<=13, -29<=k<=29, -13<=l<=12	
Reflections collected	14681	
Independent reflections	5309 [R(int) = 0.0733]	
Completeness to theta = 29.30°	98.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5309 / 0 / 248	
Goodness-of-fit on F^2	0.905	
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1213	
R indices (all data)	R1 = 0.0857, wR2 = 0.1343	
Extinction coefficient	0.0137(11)	
Largest diff. peak and hole	0.925 and -1.257 e.$Å^{-3}$	

Table 39.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcOCl(L^{5a})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	-11587(1)	-831(1)	-6607(1)	37(1)
Cl	-13533(1)	-498(1)	-8652(1)	50(1)
S(1)	-12930(1)	-1715(1)	-7044(2)	55(1)
S(9)	-10249(1)	-354(1)	-7792(1)	41(1)
O(10)	-11697(4)	-443(2)	-5176(4)	55(1)
N(3)	-10646(4)	-2209(2)	-4892(4)	43(1)
N(5)	-9827(4)	-1335(2)	-5853(4)	37(1)
N(6)	-12354(5)	-2867(2)	-6169(6)	62(1)
N(7)	-8500(4)	-1157(2)	-5945(4)	41(1)
N(51)	-7436(4)	-505(2)	-7121(5)	55(1)
C(2)	-11870(5)	-2296(2)	-5970(6)	49(1)
C(4)	-9715(5)	-1774(2)	-4803(5)	39(1)
C(8)	-8599(5)	-707(2)	-6848(5)	39(1)
C(31)	-8514(5)	-1773(2)	-3399(5)	40(1)
C(32)	-7863(5)	-1225(2)	-2757(5)	46(1)
C(33)	-6892(6)	-1227(2)	-1340(6)	57(1)
C(34)	-6532(6)	-1773(3)	-566(6)	61(1)
C(35)	-7159(5)	-2318(2)	-1210(6)	53(1)
C(36)	-8134(5)	-2318(2)	-2615(5)	45(1)
C(41)	-13800(8)	-3017(3)	-7154(10)	92(2)
C(42)	-13790(14)	-3150(5)	-8621(14)	136(4)
C(43)	-11480(5)	-3380(2)	-5359(6)	54(1)
C(44)	-10357(6)	-3581(3)	-5962(7)	66(2)
C(52)	-7417(6)	-6(3)	-8071(8)	72(2)
C(53)	-6090(7)	-779(3)	-6314(10)	88(3)

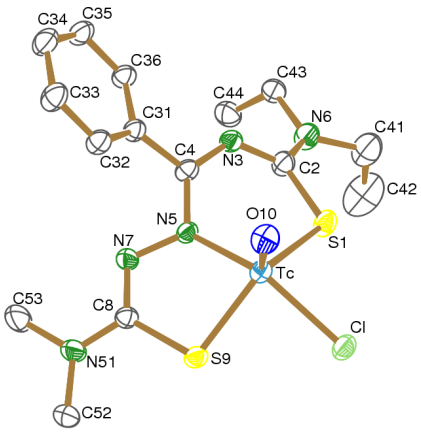

Figure 39 Ellipsoid plot (50% probability) of [TcOCl(L^{5a})].

40 [ReO(L^{5a})(Ph_2btu)], (32)

Table 40.1 Crystal data and structure refinement for [ReO(L^{5a})(Ph_2btu)].

Empirical formula	$C_{35}H_{36}N_7O_2ReS_3$	
Formula weight	869.13	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 10.381(2) Å	α= 95.65(1)°
	b = 10.897(2) Å	β= 106.44(1)°
	c = 17.526(2) Å	γ = 102.96(1)°
Volume	1825.1(5) $Å^3$	
Z	2	
Density (calculated)	1.582 g/cm^3	
Absorption coefficient	3.542 mm^{-1}	
F(000)	868	
Crystal description	Needle	
Crystal color	Dark	
Crystal size	0.270 x 0.113 x 0.020 mm^3	
Theta range for data collection	2.70 to 29.32	
Index ranges	-14<=h<=14, -14<=k<=11, -22<=l<=24	
Reflections collected	19621	
Independent reflections	9664 [R(int) = 0.1001]	
Completeness to theta = 29.32°	96.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.6498 and 0.4434	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9664 / 0 / 434	
Goodness-of-fit on F^2	0.744	
Final R indices [I>2sigma(I)]	R1 = 0.0691, wR2 = 0.1718	
R indices (all data)	R1 = 0.1255, wR2 = 0.2151	
Extinction coefficient	0.053(3)	
Largest diff. peak and hole	1.723 and -2.198 $e.Å^{-3}$	

Table 40.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^{5a})(Ph_2btu)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	686(1)	371(1)	3007(1)	49(1)
O(10)	-479(8)	-658(7)	3264(4)	58(2)
S(1)	1547(3)	1944(3)	4186(2)	59(1)
C(2)	103(12)	2565(9)	4016(6)	55(2)
N(3)	-330(9)	3069(8)	3351(5)	53(2)
C(4)	-387(11)	2638(9)	2620(6)	51(2)
N(5)	-453(8)	1415(7)	2347(4)	46(2)
N(7)	-1143(9)	1045(8)	1506(5)	54(2)
C(8)	-915(11)	35(9)	1180(5)	48(2)
S(9)	287(3)	-753(2)	1707(2)	53(1)
C(31)	-404(11)	3617(9)	2073(6)	50(2)
C(32)	-952(13)	4629(10)	2201(7)	61(3)
C(33)	-832(13)	5621(12)	1756(7)	69(3)
C(34)	-128(16)	5580(12)	1178(8)	81(4)
C(35)	434(14)	4579(11)	1062(7)	72(3)
C(36)	307(12)	3581(10)	1497(6)	58(2)
N(6)	-542(10)	2611(9)	4565(5)	60(2)
C(41)	-1880(14)	2951(14)	4369(8)	76(3)
C(42)	-3068(17)	1810(20)	3984(10)	112(6)
C(43)	-37(13)	2262(10)	5358(6)	64(3)
C(44)	970(20)	3342(14)	5974(8)	109(6)
N(51)	-1546(11)	-386(9)	391(6)	66(2)
C(52)	-1448(14)	-1560(11)	-53(7)	69(3)
C(53)	-2522(15)	274(13)	-69(8)	80(4)
S(11)	2670(3)	-388(3)	3699(2)	57(1)
C(12)	3448(10)	-600(9)	2945(6)	51(2)
N(13)	3759(9)	230(8)	2484(5)	52(2)
C(14)	3390(10)	1324(9)	2478(6)	50(2)
O(15)	2422(7)	1594(6)	2693(4)	52(2)
C(61)	4164(11)	2282(9)	2107(6)	52(2)
C(62)	3918(13)	3472(11)	2104(8)	68(3)
C(63)	4592(15)	4382(12)	1746(9)	77(4)
C(64)	5514(15)	4059(12)	1371(9)	81(4)
C(65)	5751(12)	2869(12)	1370(7)	66(3)

C(66)	5110(11)	1973(10)	1745(6)	57(2)
N(16)	3924(9)	-1664(7)	2891(5)	53(2)
C(71)	4483(11)	-1927(10)	2252(7)	56(2)
C(72)	3610(14)	-2349(12)	1475(7)	65(3)
C(73)	4160(16)	-2622(13)	862(8)	76(3)
C(74)	5584(16)	-2456(13)	1048(9)	76(4)
C(75)	6427(15)	-2064(13)	1793(10)	80(4)
C(76)	5899(13)	-1794(11)	2429(9)	69(3)
C(81)	3850(11)	-2576(9)	3428(7)	56(2)
C(82)	2893(13)	-3737(11)	3169(7)	64(3)
C(83)	2860(14)	-4596(11)	3703(8)	70(3)
C(84)	3726(14)	-4312(12)	4462(8)	72(3)
C(85)	4709(13)	-3138(12)	4733(8)	70(3)
C(86)	4774(12)	-2263(11)	4209(7)	66(3)

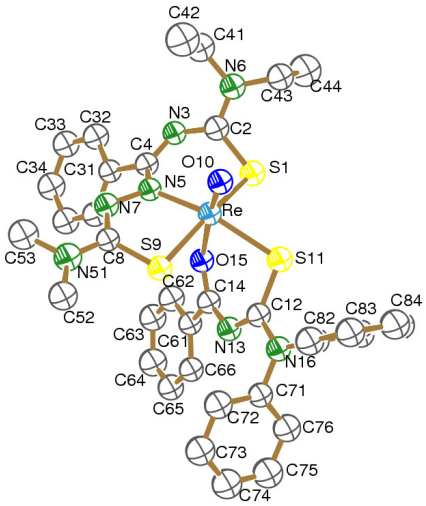

Figure 40 Ellipsoid plot (50% probability) of [ReO(L^{5a})(Ph_2btu)].

41 [Re(N)(L^{5d})(PPh_3)], (33d)

Table 41.1 Crystal data and structure refinement for [Re(N)(L^{5d})(PPh_3)].

Empirical formula	$C_{37}H_{42}N_6PReS_2$	
Formula weight	852.06	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 14.763(1) Å	α= 90°
	b = 25.552(1) Å	β= 107.03(1)°
	c = 20.082(1) Å	γ = 90°
Volume	7243.1(7) $Å^3$	
Z	8	
Density (calculated)	1.563 g/cm^3	
Absorption coefficient	3.550 mm^{-1}	
F(000)	3424	
Crystal description	Plate	
Crystal color	Orange	
Crystal size	0.320 x 0.163 x 0.040 mm^3	
Theta range for data collection	1.65 to 29.29	
Index ranges	-20<=h<=20, -35<=k<=35, -27<=l<=25	
Reflections collected	40089	
Independent reflections	9792 [R(int) = 0.0894]	
Completeness to theta = 29.29°	98.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.8647 and 0.4833	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9792 / 0 / 425	
Goodness-of-fit on F^2	1.008	
Final R indices [I>2sigma(I)]	R1 = 0.0456, wR2 = 0.1004	
R indices (all data)	R1 = 0.0687, wR2 = 0.1179	
Extinction coefficient	0.00163(7)	
Largest diff. peak and hole	1.849 and -3.059 e.$Å^{-3}$	

Table 41.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Re(N)(L^{5d})(PPh_3)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	2931(1)	5678(1)	8289(1)	29(1)
N(10)	2799(3)	5651(2)	9080(3)	41(1)
S(1)	1430(1)	5715(1)	7474(1)	41(1)
C(2)	1157(4)	6377(2)	7293(3)	42(1)
N(3)	1578(4)	6777(2)	7672(3)	46(1)
C(4)	2468(4)	6808(2)	8068(3)	41(1)
N(5)	3143(3)	6461(2)	8090(3)	36(1)
N(7)	4074(4)	6687(2)	8258(3)	44(1)
C(8)	4742(4)	6345(2)	8342(3)	38(1)
S(9)	4538(1)	5660(1)	8372(1)	33(1)
C(31)	2698(5)	7297(2)	8501(4)	48(1)
C(32)	2268(5)	7775(3)	8246(4)	54(2)
C(33)	2429(6)	8208(3)	8675(5)	67(2)
C(34)	2980(7)	8174(3)	9352(5)	78(3)
C(35)	3404(8)	7712(4)	9605(5)	90(3)
C(36)	3262(7)	7272(3)	9175(4)	69(2)
N(6)	409(3)	6492(2)	6742(3)	45(1)
C(41)	188(5)	7041(3)	6549(4)	57(2)
C(42)	804(6)	7265(3)	6150(5)	71(2)
C(43)	-160(4)	6085(3)	6310(4)	59(2)
C(44)	217(7)	5903(4)	5712(5)	83(3)
N(51)	5649(3)	6508(2)	8421(3)	45(1)
C(52)	5861(5)	7067(3)	8463(5)	63(2)
C(53)	6019(7)	7300(3)	9187(6)	79(3)
C(54)	6432(7)	6943(3)	9788(5)	77(3)
C(55)	7226(7)	6591(4)	9754(5)	78(2)
C(56)	6935(5)	6081(3)	9349(4)	55(2)
C(57)	6463(4)	6158(2)	8581(3)	42(1)
P	2878(1)	4755(1)	8068(1)	30(1)
C(61)	1927(4)	4385(2)	8260(3)	39(1)
C(62)	1775(5)	3864(3)	8052(4)	52(2)
C(63)	1078(6)	3575(3)	8225(5)	68(2)
C(64)	570(5)	3799(4)	8633(5)	76(3)
C(65)	737(5)	4306(4)	8848(5)	74(3)

C(66)	1405(4)	4601(3)	8657(4)	50(2)
C(71)	2726(3)	4661(2)	7145(3)	31(1)
C(72)	1838(4)	4565(3)	6682(3)	41(1)
C(73)	1726(5)	4557(3)	5975(3)	51(2)
C(74)	2486(5)	4638(3)	5716(3)	47(1)
C(75)	3363(5)	4740(2)	6173(3)	43(1)
C(76)	3490(4)	4755(2)	6883(3)	35(1)
C(81)	3900(3)	4368(2)	8522(3)	32(1)
C(82)	4256(4)	3960(2)	8212(3)	42(1)
C(83)	5001(5)	3656(3)	8601(4)	51(2)
C(84)	5393(4)	3749(3)	9302(4)	54(2)
C(85)	5042(4)	4146(3)	9616(3)	48(1)
C(86)	4311(4)	4460(2)	9237(3)	40(1)

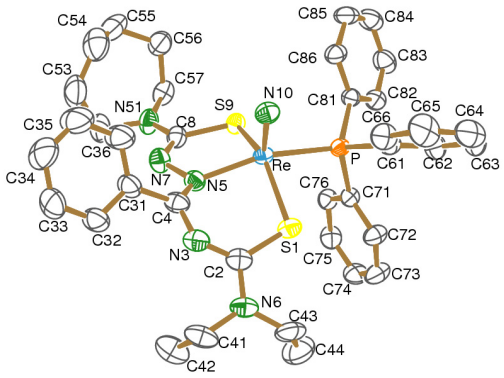

Figure 41 Ellipsoid plot (50% probability) of [Re(N)(L^{5d})(PPh_3)].

42 [ReO(L^{6a})(ReO_4)], (34)

Table 42.1 Crystal data and structure refinement for [ReO(L^{6a})(ReO_4)].

Empirical formula	$C_{30}H_{34}N_6O_5Re_2S_2$	
Formula weight	995.15	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 14.109(1) Å	α= 90°
	b = 11.160(1) Å	β= 102.96(1)°
	c = 21.778(2) Å	γ = 90°
Volume	3341.8(5) $Å^3$	
Z	4	
Density (calculated)	1.978 g/cm^3	
Absorption coefficient	7.410 mm^{-1}	
F(000)	1912	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.4 x 0.2 x 0.2 mm^3	
Theta range for data collection	2.06 to 29.22	
Index ranges	-19<=h<=19, -14<=k<=15, -29<=l<=24	
Reflections collected	34810	
Independent reflections	8904 [R(int) = 0.1104]	
Completeness to theta = 29.22°	98.2 %	
Absorption correction	Integration	
Max. and min. transmission	0.5167 and 0.2169	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8904 / 0 / 398	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0762, wR2 = 0.1812	
R indices (all data)	R1 = 0.1169, wR2 = 0.2003	
Largest diff. peak and hole	8.776 and -4.817 $e.Å^{-3}$	

Table 42.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [ReO(L^{6a})(ReO_4)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	1050(1)	7803(1)	4429(1)	34(1)
O(10)	32(5)	8322(7)	4627(4)	44(2)
O(20)	2449(7)	7120(9)	4144(7)	74(4)
S(1)	370(3)	6886(3)	3451(2)	50(1)
C(2)	755(9)	7875(11)	2930(6)	45(3)
N(3)	653(7)	9071(9)	2977(5)	43(2)
C(4)	928(8)	9727(11)	3473(6)	43(3)
N(5)	1475(7)	9288(8)	4022(5)	39(2)
C(31)	603(9)	10984(10)	3422(6)	43(3)
C(32)	490(8)	11615(11)	3958(7)	45(3)
C(33)	127(10)	12773(11)	3895(8)	58(4)
C(34)	-134(12)	13314(14)	3324(10)	73(5)
C(35)	-21(18)	12709(16)	2774(11)	91(6)
C(36)	352(13)	11517(13)	2837(8)	66(4)
N(6)	1108(8)	7426(10)	2472(5)	49(2)
C(41)	1206(10)	6147(12)	2363(7)	55(3)
C(42)	321(12)	5673(15)	1882(9)	69(4)
C(43)	1457(15)	8205(14)	2048(8)	72(5)
C(44)	2535(17)	8407(19)	2233(15)	114(9)
C(51)	2279(8)	9912(9)	4404(6)	38(2)
C(52)	2793(8)	10840(11)	4203(6)	43(3)
C(53)	3603(8)	11290(11)	4591(7)	49(3)
C(54)	3954(8)	10839(10)	5186(7)	44(3)
C(55)	3473(7)	9870(10)	5394(6)	42(3)
C(56)	2622(7)	9439(9)	5018(6)	37(2)
S(11)	1133(2)	5831(2)	4829(2)	45(1)
C(12)	2142(7)	5918(8)	5464(6)	38(2)
N(13)	2256(7)	6840(8)	5881(5)	43(2)
C(14)	2217(7)	7972(9)	5740(6)	37(2)
N(15)	2104(6)	8419(8)	5141(5)	38(2)
C(61)	2267(7)	8805(10)	6286(6)	39(2)
C(62)	1767(8)	9893(11)	6202(7)	47(3)
C(63)	1833(9)	10659(11)	6704(7)	53(3)
C(64)	2357(10)	10374(14)	7309(8)	60(4)

C(65)	2836(10)	9267(13)	7392(7)	56(3)
C(66)	2785(9)	8477(10)	6876(6)	45(3)
N(16)	2763(8)	5039(9)	5594(7)	57(3)
C(71)	3675(15)	5133(17)	6065(12)	110(10)
C(72)	4380(20)	5820(40)	6010(20)	200(20)
C(73)	2661(9)	3928(11)	5203(8)	56(3)
C(74)	2085(15)	2990(13)	5462(14)	99(8)
Re(2)	3583(1)	6883(1)	4046(1)	77(1)
O(30)	3989(18)	8010(30)	3683(11)	223(16)
O(50)	4351(18)	6910(30)	4738(19)	280(30)
O(40)	3459	5997	3414	207(11)

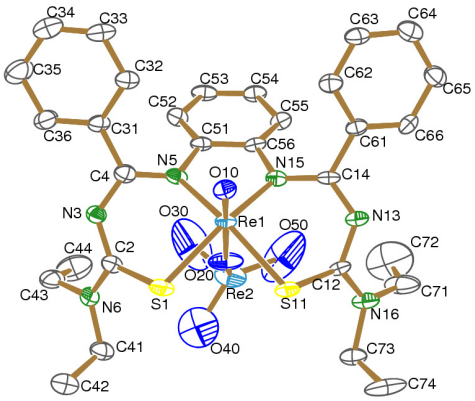

Figure 42 Ellipsoid plot (40% probability) of [ReO(L^{6a})(ReO_4)].

43 [{ReO(L^{6b})}$_2$O], (35)

Table 43.1 Crystal data and structure refinement for [{ReO(L^{6b})}$_2$O] · EtOH.

Empirical formula	$C_{62}H_{66}N_{12}O_8Re_2S_4$	
Formula weight	1607.91	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	$P4_12_12$	
Unit cell dimensions	a = 19.549(1) Å	α= 90°
	b = 19.549(1) Å	β= 90°
	c = 17.943(1) Å	γ = 90°
Volume	6856.8(6) Å^3	
Z	4	
Density (calculated)	1.558 g/cm^3	
Absorption coefficient	3.709 mm^{-1}	
F(000)	3200	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.100 mm^3	
Theta range for data collection	1.54 to 26.81	
Index ranges	-22<=h<=24, -24<=k<=18, -22<=l<=22	
Reflections collected	26724	
Independent reflections	7291 [R(int) = 0.2020]	
Completeness to theta = 26.81°	99.4 %	
Absorption correction	Integration	
Max. and min. transmission	0.7579 and 0.2728	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7291 / 0 / 402	
Goodness-of-fit on F^2	0.977	
Final R indices [I>2sigma(I)]	R1 = 0.0717, wR2 = 0.1675	
R indices (all data)	R1 = 0.1007, wR2 = 0.1862	
Absolute structure parameter	0.01(2)	
Extinction coefficient	0.0036(3)	
Largest diff. peak and hole	4.270 and -2.287 e.Å^{-3}	

Table 43.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{ReO(L^{6b})}$_2$O] · EtOH. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re(1)	1811(1)	7338(1)	8343(1)	35(1)
O(10)	1440(5)	6965(6)	9126(7)	49(2)
O(20)	2264(5)	7736(5)	7500	30(2)
S(1)	1157(2)	8360(2)	8432(3)	52(1)
C(2)	1720(11)	8905(9)	8868(12)	65(5)
N(3)	2162(7)	8753(8)	9414(9)	59(4)
C(4)	2508(7)	8169(9)	9499(8)	47(4)
N(5)	2631(6)	7714(6)	8950(6)	38(2)
C(31)	2740(9)	8068(12)	10272(9)	64(6)
C(32)	2747(11)	7410(12)	10572(9)	65(5)
C(33)	2883(13)	7337(15)	11298(10)	86(7)
C(34)	3042(15)	7909(16)	11744(12)	98(8)
C(35)	3053(15)	8543(15)	11472(12)	96(9)
C(36)	2916(10)	8646(11)	10712(9)	62(5)
N(6)	1664(9)	9610(8)	8730(12)	75(5)
C(41)	1100(12)	9917(13)	8269(19)	97(8)
C(42)	692(17)	10341(15)	8761(18)	122(12)
O(43)	1133(14)	10859(11)	9099(16)	158(11)
C(44)	1600(20)	10470(20)	9690(30)	220(30)
C(45)	2060(12)	10099(12)	9136(18)	95(8)
C(51)	3223(7)	7305(8)	8923(6)	36(3)
C(52)	3853(7)	7509(8)	9189(8)	43(4)
C(53)	4418(8)	7108(8)	9076(7)	37(3)
C(54)	4362(8)	6512(10)	8693(8)	48(4)
C(55)	3759(7)	6301(8)	8386(8)	38(3)
C(56)	3158(7)	6674(7)	8518(6)	36(3)
S(11)	1015(2)	6886(2)	7472(2)	40(1)
C(12)	1325(7)	6073(7)	7278(8)	33(3)
N(13)	1780(6)	5694(6)	7631(6)	36(3)
C(14)	2297(8)	5891(8)	8068(7)	39(3)
N(15)	2512(5)	6517(6)	8236(5)	30(2)
C(131)	2670(8)	5273(6)	8396(8)	39(3)
C(132)	2786(8)	5260(9)	9169(9)	50(4)
C(133)	3124(13)	4672(11)	9466(10)	73(6)

C(134)	3282(12)	4135(9)	9042(10)	64(5)
C(135)	3153(11)	4154(9)	8266(11)	64(5)
C(136)	2811(9)	4694(8)	7976(9)	50(4)
N(16)	1030(7)	5740(7)	6687(8)	48(3)
C(141)	514(9)	6026(9)	6208(10)	53(4)
C(142)	-122(10)	5591(10)	6189(9)	61(5)
O(143)	43(7)	4916(6)	5984(7)	59(3)
C(144)	545(9)	4614(9)	6493(10)	54(4)
C(145)	1187(9)	5022(9)	6514(11)	57(5)
C(61)	-1014(11)	3645(11)	6719(12)	22(4)
C(60)	-1420(20)	4180(20)	6460(20)	68(10)
O(61)	-1768(13)	4459(10)	5754(14)	58(7)

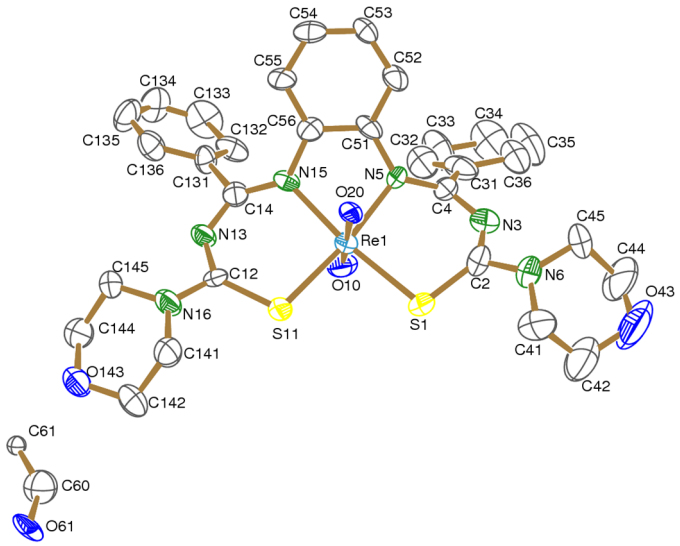

Figure 43 Ellipsoid plot (50% probability) of the asymmetric unit of [{ReO(L^{6b})}$_2$O] · EtOH.

44 [Re(N)(L^{6b})], (36)

Table 44.1 Crystal data and structure refinement for [Re(N)(L^{6b})].

Empirical formula	$C_{30}H_{30}N_7O_2ReS_2$	
Formula weight	770.93	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.958(1) Å	α= 90°
	b = 18.620(1) Å	β= 102.02(1)°
	c = 15.813(2) Å	γ = 90°
Volume	2867.8(5) $Å^3$	
Z	4	
Density (calculated)	1.786 g/cm^3	
Absorption coefficient	4.426 mm^{-1}	
F(000)	1528	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.100 mm^3	
Theta range for data collection	2.85 to 29.33	
Index ranges	-13<=h<=13, -22<=k<=25, -21<=l<=18	
Reflections collected	15349	
Independent reflections	7593 [R(int) = 0.0575]	
Completeness to theta = 29.33°	96.6 %	
Absorption correction	Integration	
Max. and min. transmission	0.4901 and 0.1662	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7593 / 0 / 433	
Goodness-of-fit on F^2	1.023	
Final R indices [I>2sigma(I)]	R1 = 0.0595, wR2 = 0.1386	
R indices (all data)	R1 = 0.0963, wR2 = 0.1567	
Largest diff. peak and hole	3.454 and -2.764 $e.Å^{-3}$	

Table 44.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Re(N)(L^{6b})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	8973(1)	8765(1)	7571(1)	46(1)
N(10)	7932(7)	8846(4)	6617(5)	57(2)
S(1)	7949(2)	7868(1)	8269(2)	59(1)
C(2)	8895(9)	7089(5)	8201(7)	63(2)
N(3)	9461(8)	6939(4)	7524(5)	56(2)
C(4)	10160(8)	7387(4)	7119(6)	46(2)
N(5)	10500(6)	8067(4)	7365(5)	47(2)
C(31)	10524(8)	7061(4)	6339(6)	51(2)
C(32)	10474(8)	7444(5)	5595(6)	55(2)
C(33)	10652(10)	7118(5)	4853(8)	70(3)
C(34)	10979(11)	6390(6)	4860(8)	74(3)
C(35)	11096(12)	5987(7)	5603(9)	80(3)
C(36)	10862(10)	6326(5)	6340(8)	64(2)
N(6)	9100(20)	6669(10)	8917(15)	56(5)
C(41)	7790(20)	6455(10)	9250(16)	57(5)
C(42)	8430(20)	6070(9)	10019(15)	67(5)
O(43)	8846(16)	5386(7)	9871(11)	69(4)
C(44)	9840(20)	5395(11)	9350(18)	80(7)
C(45)	9200(30)	5760(12)	8440(20)	71(7)
N(6A)	8680(20)	6462(9)	8604(13)	49(4)
C(41A)	9630(30)	5926(12)	8818(16)	57(5)
C(42A)	8440(20)	5424(11)	8670(20)	86(8)
O(43A)	7730(20)	5467(8)	9371(16)	104(7)
C(44A)	7170(20)	6169(10)	9380(20)	84(8)
C(45A)	8320(30)	6701(12)	9588(14)	60(5)
C(51)	11757(7)	8386(4)	7297(6)	47(2)
C(52)	12933(9)	8022(5)	7173(8)	64(3)
C(53)	14154(9)	8382(6)	7232(8)	74(3)
C(54)	14257(10)	9109(6)	7417(8)	71(3)
C(55)	13105(8)	9474(5)	7515(7)	59(2)
C(56)	11831(8)	9127(5)	7433(6)	50(2)
S(11)	8071(2)	9565(1)	8464(2)	53(1)
C(12)	9013(9)	10363(5)	8500(6)	56(2)
N(13)	9698(7)	10571(3)	7908(5)	54(2)

C(14)	10382(8)	10166(4)	7454(6)	49(2)
N(15)	10627(7)	9451(4)	7580(5)	50(2)
C(131)	10916(8)	10568(4)	6780(6)	48(2)
C(132)	11053(8)	10246(4)	6012(6)	51(2)
C(133)	11548(9)	10634(5)	5387(7)	60(2)
C(134)	11922(9)	11347(5)	5541(7)	61(2)
C(135)	11780(11)	11669(5)	6282(8)	69(3)
C(136)	11278(10)	11291(4)	6908(7)	60(2)
N(16)	8979(8)	10818(4)	9159(6)	68(2)
C(141)	8289(12)	10662(6)	9887(8)	79(3)
C(142)	7661(15)	11297(7)	10155(10)	96(4)
O(143)	8507(11)	11905(5)	10293(7)	114(4)
C(144)	9030(19)	12070(7)	9549(12)	122(6)
C(145)	9813(13)	11484(7)	9281(11)	97(5)

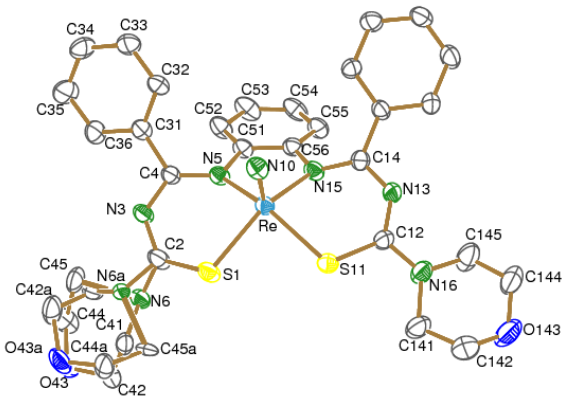

Figure 44 Ellipsoid plot (50% probability) of [Re(N)(L^{6b})].

45 [Tc(N)(L^{6b})], (37)

Table 45.1 Crystal data and structure refinement for [Tc(N)(L^{6b})].

Empirical formula	$C_{30}H_{30}N_7O_2S_2Tc$	
Formula weight	682.73	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.974(1) Å	α= 90°
	b = 18.605(2) Å	β= 102.12(1)°
	c = 15.815(1) Å	γ = 90°
Volume	2869.5(5) $Å^3$	
Z	4	
Density (calculated)	1.580 g/cm^3	
Absorption coefficient	0.690 mm^{-1}	
F(000)	1400	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.25 x 0.15 x 0.10 mm^3	
Theta range for data collection	1.71 to 29.29	
Index ranges	-10<=h<=13, -25<=k<=25, -21<=l<=21	
Reflections collected	19021	
Independent reflections	7723 [R(int) = 0.1457]	
Completeness to theta = 29.29°	98.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7723 / 0 / 417	
Goodness-of-fit on F^2	0.954	
Final R indices [I>2sigma(I)]	R1 = 0.0834, wR2 = 0.1785	
R indices (all data)	R1 = 0.1984, wR2 = 0.2566	
Extinction coefficient	0.0013(6)	
Largest diff. peak and hole	0.780 and -0.942 $e.Å^{-3}$	

Table 45.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Tc(N)(L^{6b})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	8981(1)	8771(1)	7556(1)	34(1)
N(10)	7966(9)	8854(4)	6630(5)	49(2)
S(1)	7980(3)	7877(1)	8278(2)	50(1)
C(2)	8879(13)	7090(5)	8173(7)	55(3)
N(3)	9491(9)	6939(4)	7514(5)	42(2)
C(4)	10187(10)	7377(5)	7127(6)	40(2)
N(5)	10526(8)	8067(4)	7364(5)	37(2)
C(31)	10566(9)	7048(5)	6354(6)	37(2)
C(32)	10508(11)	7444(5)	5610(6)	47(2)
C(33)	10699(14)	7107(6)	4875(7)	63(3)
C(34)	10992(14)	6394(6)	4866(8)	66(3)
C(35)	11123(16)	5997(6)	5620(8)	76(4)
C(36)	10888(13)	6333(5)	6365(7)	56(3)
N(6)	8905(13)	6573(6)	8768(8)	88(4)
C(41)	9460(20)	5857(8)	8660(14)	125(9)
C(42)	9780(30)	5404(12)	9305(15)	65(8)
O(43)	8760(20)	5354(7)	9836(11)	57(5)
C(44)	8440(30)	6065(12)	10022(14)	53(6)
C(45)	7780(30)	6476(13)	9247(16)	50(6)
C(42A)	8460(20)	5455(11)	8669(16)	56(6)
O(43A)	7740(20)	5476(9)	9334(15)	73(6)
C(44A)	7330(40)	6171(13)	9450(20)	90(13)
C(45A)	8440(30)	6709(12)	9620(15)	44(5)
C(51)	11883(9)	9142(5)	7439(5)	36(2)
C(52)	13106(12)	9488(6)	7520(6)	52(3)
C(53)	14269(12)	9107(7)	7421(7)	53(3)
C(54)	14189(15)	8381(7)	7220(8)	66(3)
C(55)	12972(10)	8039(6)	7181(6)	48(2)
C(56)	11745(10)	8399(5)	7280(6)	39(2)
S(11)	8106(3)	9563(1)	8474(2)	44(1)
C(12)	8972(11)	10363(5)	8483(6)	44(2)
N(13)	9657(10)	10574(4)	7886(5)	48(2)
C(14)	10397(11)	10170(5)	7460(5)	40(2)
N(15)	10599(8)	9456(4)	7574(4)	36(2)

C(131)	10934(10)	10569(5)	6783(5)	37(2)
C(132)	11103(10)	10263(5)	6017(5)	42(2)
C(133)	11514(10)	10650(6)	5393(6)	47(2)
C(134)	11893(12)	11338(6)	5539(6)	57(3)
C(135)	11754(14)	11679(6)	6284(7)	60(3)
C(136)	11263(11)	11296(6)	6901(6)	51(2)
N(16)	8953(10)	10824(5)	9132(6)	57(2)
C(141)	8328(13)	10674(8)	9888(7)	67(3)
C(142)	7629(17)	11317(8)	10117(9)	81(4)
O(143)	8413(15)	11919(6)	10217(8)	120(5)
C(144)	9020(30)	12088(8)	9514(13)	155(12)
C(145)	9806(19)	11511(8)	9260(12)	96(5)

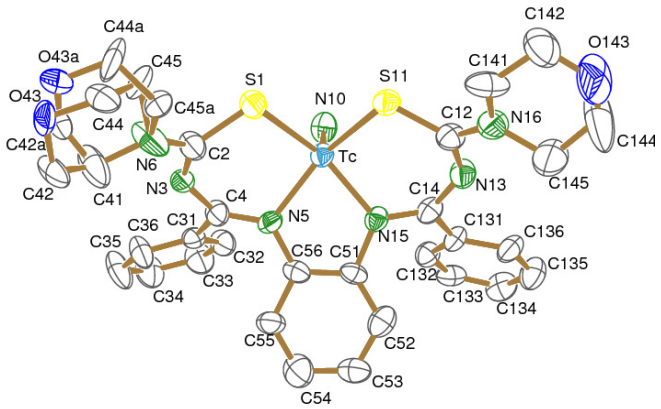

Figure 45 Ellipsoid plot (50% probability) of [Tc(N)(L^{6b})]

46 [ReO(OMe)(L^7)], (38)

Table 46.1 Crystal data and structure refinement for [ReO(OMe)(L^7)].

Empirical formula	$C_{23}H_{28}N_6O_2ReS_2$	
Formula weight	671.86	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.927(1) Å	α= 79.89(1)°
	b = 10.623(1) Å	β= 79.07(1)°
	c = 13.863(1) Å	γ = 64.31(1)°
Volume	1286.3(2) $Å^3$	
Z	2	
Density (calculated)	1.732 g/cm^3	
Absorption coefficient	4.918 mm^{-1}	
F(000)	662	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.5 x 0.5 x 0.1 mm^3	
Theta range for data collection	2.14 to 29.21	
Index ranges	-13<=h<=13, -14<=k<=14, -18<=l<=18	
Reflections collected	23666	
Independent reflections	6895 [R(int) = 0.0824]	
Completeness to theta = 29.21°	99.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.7682 and 0.3537	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6895 / 0 / 316	
Goodness-of-fit on F^2	1.046	
Final R indices [I>2sigma(I)]	R1 = 0.0379, wR2 = 0.0800	
R indices (all data)	R1 = 0.0508, wR2 = 0.0890	
Largest diff. peak and hole	3.535 and -2.341 $e.Å^{-3}$	

Table 46.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(OMe)(L^7)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	5505(1)	6633(1)	6902(1)	24(1)
O(10)	5233(4)	5127(4)	7223(3)	34(1)
O(20)	5562(4)	8425(4)	6456(3)	32(1)
C(20B)	5473(18)	9623(14)	6798(12)	56(4)
C(20A)	5390(20)	9462(18)	5678(12)	71(5)
S(1)	6629(1)	6485(2)	8296(1)	34(1)
C(2)	5096(6)	7306(6)	9172(4)	34(1)
N(3)	3857(5)	8440(5)	8954(3)	34(1)
C(4)	3084(5)	8710(5)	8217(3)	28(1)
N(5)	3324(5)	7828(4)	7549(3)	28(1)
C(31)	1819(6)	10142(6)	8145(4)	32(1)
C(32)	1367(7)	10811(6)	7225(4)	39(1)
C(33)	193(8)	12126(7)	7156(5)	48(2)
C(34)	-513(8)	12798(7)	7993(6)	56(2)
C(35)	-44(8)	12167(7)	8897(6)	56(2)
C(36)	1124(7)	10854(7)	8977(4)	42(1)
N(6)	5203(6)	6831(6)	10125(3)	44(1)
C(41)	6579(7)	5625(9)	10468(4)	52(2)
C(42)	7817(10)	6057(11)	10454(5)	67(2)
C(43)	3949(8)	7506(10)	10886(5)	69(3)
C(44)	4103(13)	8663(16)	11296(9)	126(6)
C(51)	2068(6)	7816(5)	7225(4)	31(1)
C(52)	818(6)	7919(7)	7935(5)	41(1)
C(53)	-484(7)	7951(7)	7691(6)	50(2)
C(54)	-547(8)	7830(8)	6741(6)	56(2)
C(55)	675(7)	7677(7)	6030(5)	45(1)
C(56)	2021(6)	7654(6)	6248(4)	34(1)
C(7)	3251(6)	7446(6)	5408(4)	30(1)
C(8)	2859(7)	7749(7)	4368(4)	44(1)
N(9)	4672(5)	6956(5)	5533(3)	28(1)
N(10)	5682(5)	6677(5)	4669(3)	31(1)
C(11)	7114(6)	6060(5)	4784(3)	28(1)
S(12)	7862(1)	5607(2)	5907(1)	32(1)
N(13)	8123(5)	5704(6)	3974(3)	37(1)

C(14)	7687(8)	5927(8)	3002(4)	46(2)

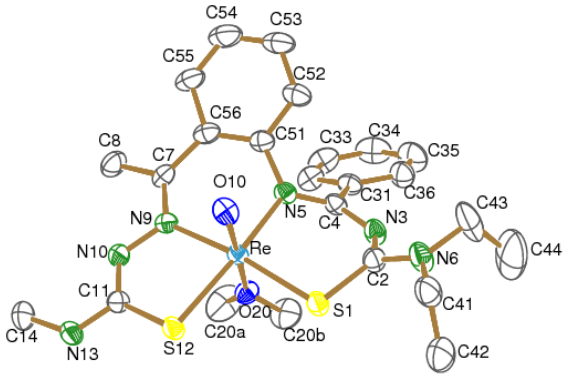

Figure 46 Ellipsoid plot (50% probability) of [ReO(OMe)(L^7)].

47 [ReN(L^7)], (39)

Table 47.1 Crystal data and structure refinement for [ReN(L^7)].

Empirical formula	$C_{22}H_{26}N_7ReS_2$	
Formula weight	638.82	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 8.598(1) Å	α= 66.64(1)°
	b = 10.974(1) Å	β= 79.86(1)°
	c = 13.669(1) Å	γ = 77.99(1)°
Volume	1151.8 (2) $Å^3$	
Z	2	
Density (calculated)	1.842 g/cm^3	
Absorption coefficient	5.482 mm^{-1}	
F(000)	628	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.300 x 0.250 x 0.100 mm^3	
Theta range for data collection	2.05 to 29.20	
Index ranges	-11<=h<=11, -14<=k<=14, -18<=l<=18	
Reflections collected	21421	
Independent reflections	6186 [R(int) = 0.1379]	
Completeness to theta = 29.20°	99.2 %	
Absorption correction	Integration	
Max. and min. transmission	0.3541 and 0.2036	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6186 / 0 / 289	
Goodness-of-fit on F^2	1.157	
Final R indices [I>2sigma(I)]	R1 = 0.0357, wR2 = 0.0901	
R indices (all data)	R1 = 0.0389, wR2 = 0.0978	
Largest diff. peak and hole	2.721 and -4.496 $e.Å^{-3}$	

Table 47.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReN(L^7)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	7838(1)	6917(1)	7106(1)	22(1)
N	6148(5)	6604(5)	6877(3)	32(1)
S(1)	8451(1)	5278(1)	8778(1)	28(1)
C(2)	7582(5)	6013(5)	9706(3)	27(1)
N(3)	7712(5)	7278(4)	9552(3)	29(1)
C(4)	7625(5)	8338(5)	8666(3)	24(1)
N(5)	7205(4)	8404(4)	7719(3)	24(1)
C(31)	8048(5)	9552(5)	8726(3)	25(1)
C(32)	8897(6)	10415(5)	7859(4)	31(1)
C(33)	9380(7)	11505(6)	7940(4)	38(1)
C(34)	9005(8)	11732(6)	8892(6)	45(1)
C(35)	8159(8)	10858(7)	9762(5)	46(1)
C(36)	7687(6)	9787(6)	9671(4)	34(1)
N(6)	6931(6)	5276(5)	10660(3)	38(1)
C(41)	6575(8)	5807(7)	11546(5)	44(1)
C(42)	4912(8)	6636(8)	11497(6)	53(2)
C(43)	6681(6)	3878(5)	10939(4)	34(1)
C(44)	8068(8)	2858(6)	11474(4)	43(1)
C(7)	7580(5)	9712(5)	5315(3)	25(1)
C(8)	7909(6)	10690(5)	4194(3)	33(1)
N(9)	8387(4)	8498(4)	5642(3)	24(1)
N(10)	9610(5)	8266(4)	4876(3)	29(1)
C(11)	10445(5)	7063(5)	5189(4)	29(1)
S(12)	10051(2)	5749(1)	6412(1)	33(1)
N(13)	11732(6)	6839(5)	4528(4)	41(1)
C(14)	12792(7)	5567(8)	4710(5)	48(1)
C(51)	6172(5)	9572(5)	7162(3)	24(1)
C(56)	6291(5)	10154(4)	6028(3)	23(1)
C(55)	5140(6)	11272(5)	5551(4)	29(1)
C(54)	3923(6)	11781(6)	6152(4)	35(1)
C(53)	3817(6)	11198(6)	7265(4)	36(1)
C(52)	4930(5)	10111(5)	7751(4)	31(1)

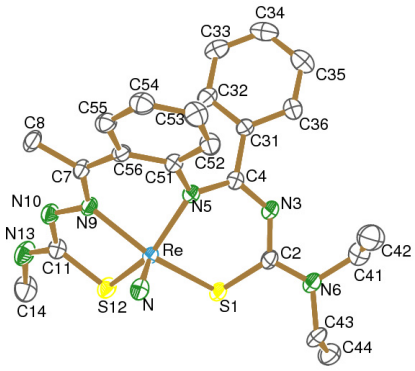

Figure 47 Ellipsoid plot (50% probability) of [ReN(L^7)].

48 [TcN(L^7)], (40)

Table 48.1 Crystal data and structure refinement for [TcN(L^7)].

Empirical formula	$C_{22}H_{26}N_7S_2Tc$	
Formula weight	550.62	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 8.577(1) Å	α= 66.82(1)°
	b = 10.974(1) Å	β= 80.34(1)°
	c = 13.672(1) Å	γ = 78.64(1)°
Volume	1153.9(1) Å^3	
Z	2	
Density (calculated)	1.585 g/cm^3	
Absorption coefficient	0.830 mm^{-1}	
F(000)	564	
Crystal description	Block	
Crystal color	Red	
Crystal size	0.20 x 0.15 x 0.10 mm^3	
Theta range for data collection	2.79 to 29.25	
Index ranges	-9<=h<=11, -15<=k<=15, -18<=l<=18	
Reflections collected	11967	
Independent reflections	6149 [R(int) = 0.0957]	
Completeness to theta = 29.25°	97.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6149 / 0 / 290	
Goodness-of-fit on F^2	0.902	
Final R indices [I>2sigma(I)]	R1 = 0.0580, wR2 = 0.0998	
R indices (all data)	R1 = 0.1224, wR2 = 0.1190	
Extinction coefficient	0.0046(8)	
Largest diff. peak and hole	0.982 and -1.120 e.Å^{-3}	

Table 48.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcN(L^7)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	7823(1)	6918(1)	7102(1)	23(1)
N	6191(6)	6586(4)	6880(3)	33(1)
S(1)	8433(2)	5282(1)	8783(1)	31(1)
C(2)	7611(7)	6016(5)	9698(4)	30(1)
N(3)	7762(6)	7279(4)	9553(3)	28(1)
C(4)	7644(6)	8336(4)	8665(4)	25(1)
N(5)	7211(5)	8394(3)	7727(3)	24(1)
C(7)	7591(7)	9709(4)	5320(4)	26(1)
C(8)	7967(8)	10668(5)	4198(4)	34(1)
N(9)	8391(5)	8489(4)	5641(3)	27(1)
N(10)	9580(6)	8249(4)	4887(3)	30(1)
C(11)	10426(7)	7049(5)	5207(4)	28(1)
S(12)	10054(2)	5753(1)	6431(1)	35(1)
N(13)	11679(6)	6811(5)	4548(4)	42(1)
C(14)	12746(9)	5576(6)	4730(5)	50(2)
C(31)	8075(7)	9557(5)	8724(4)	26(1)
C(32)	7686(7)	9792(5)	9665(4)	33(1)
C(33)	8152(8)	10866(5)	9767(5)	42(2)
C(34)	9002(8)	11741(5)	8900(5)	45(2)
C(35)	9389(7)	11515(5)	7967(5)	39(1)
C(36)	8933(7)	10421(5)	7867(4)	32(1)
N(6)	6989(6)	5286(4)	10663(3)	33(1)
C(41)	6677(8)	3909(5)	10944(4)	37(1)
C(42)	8025(8)	2880(5)	11484(5)	44(2)
C(43)	6589(9)	5835(6)	11522(5)	46(2)
C(44)	4953(9)	6641(7)	11498(6)	53(2)
C(51)	6201(6)	9562(4)	7149(4)	25(1)
C(52)	4963(7)	10102(5)	7741(4)	32(1)
C(53)	3878(7)	11189(5)	7250(4)	33(1)
C(54)	3976(7)	11770(5)	6145(5)	36(1)
C(55)	5180(7)	11262(5)	5560(4)	30(1)
C(56)	6333(6)	10141(4)	6032(4)	23(1)

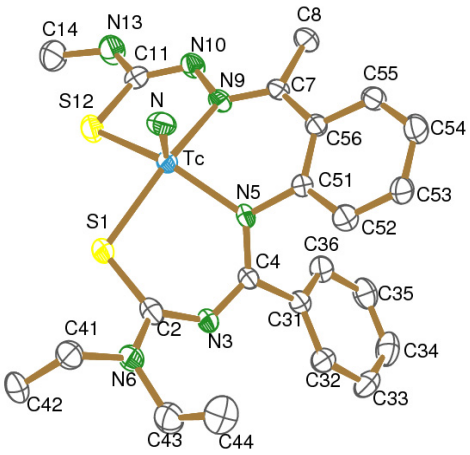

Figure 48 Ellipsoid plot (50% probability) of [TcN(L^7)].

49 [TcN(PPh$_3$)(Et$_2$tu)(L^{7b})], (41)

Table 49.1 Crystal data and structure refinement for [TcN(PPh$_3$)(Et$_2$tu)(L^{7b})] · H$_2$O.

Empirical formula	$C_{40}H_{41}N_7OPS_2Tc$	
Formula weight	828.89	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 11.622(3) Å	α= 92.45(2)°
	b = 13.182(3) Å	β= 92.73(2)°
	c = 14.620(4) Å	γ = 110.76(2)°
Volume	2087.5(9) Å^3	
Z	2	
Density (calculated)	1.319 g/cm^3	
Absorption coefficient	0.522 mm^{-1}	
F(000)	856	
Crystal description	Block	
Crystal color	Yellow	
Crystal size	0.20 x 0.13 x 0.10 mm^3	
Theta range for data collection	1.88 to 29.30	
Index ranges	-16<=h<=15, -16<=k<=18, -20<=l<=18	
Reflections collected	21440	
Independent reflections	11079 [R(int) = 0.1453]	
Completeness to theta = 29.30°	97.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11079 / 0 / 465	
Goodness-of-fit on F^2	0.865	
Final R indices [I>2sigma(I)]	R1 = 0.0714, wR2 = 0.1682	
R indices (all data)	R1 = 0.1755, wR2 = 0.2124	
Extinction coefficient	0.0210(14)	
Largest diff. peak and hole	1.546 and -0.735 e.Å^{-3}	

Table 49.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcN(PPh_3)(Et_2tu)(L^{7b})]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	7164(1)	7082(1)	5615(1)	37(1)
N	5740(5)	6393(5)	5315(4)	48(2)
S(1)	7563(2)	9002(2)	5743(1)	48(1)
C(2)	8142(6)	8880(6)	4672(5)	42(2)
N(3)	7972(6)	7858(5)	4475(4)	45(1)
N(6)	8672(6)	9713(5)	4161(4)	47(2)
C(41)	8857(8)	10848(6)	4462(5)	53(2)
C(42)	8025(8)	11318(7)	3945(6)	60(2)
C(43)	9041(8)	9477(7)	3239(5)	56(2)
C(44)	7973(9)	8994(7)	2542(5)	68(2)
C(4)	11035(7)	7187(6)	2952(5)	44(2)
N(5)	10827(6)	7303(5)	2072(4)	48(2)
C(7)	8984(7)	5739(6)	2980(4)	42(2)
C(8)	9209(8)	4694(6)	2695(5)	54(2)
N(9)	10124(5)	6543(5)	3427(4)	42(1)
N(10)	10077(5)	6641(5)	4399(4)	41(1)
C(11)	8939(7)	6074(5)	4540(4)	39(2)
S(12)	8368(2)	5955(2)	5627(1)	42(1)
N(13)	8179(5)	5611(5)	3753(4)	41(1)
C(14)	7093(7)	4618(7)	3791(6)	59(2)
C(31)	12270(7)	7741(6)	3416(5)	45(2)
C(32)	12994(8)	8743(6)	3141(6)	56(2)
C(33)	14149(8)	9245(7)	3564(8)	76(3)
C(34)	14609(8)	8765(8)	4230(6)	66(2)
C(35)	13876(8)	7768(8)	4493(5)	61(2)
C(36)	12718(7)	7254(6)	4096(5)	47(2)
C(51)	9601(7)	6971(6)	1717(5)	48(2)
C(52)	9324(9)	7394(7)	887(5)	58(2)
C(53)	8127(9)	7126(8)	549(5)	62(2)
C(54)	7171(8)	6448(8)	1013(5)	63(2)
C(55)	7401(8)	6022(7)	1811(5)	57(2)
C(56)	8612(7)	6251(6)	2165(4)	44(2)
P	6949(2)	6802(2)	7223(1)	39(1)
C(61)	5691(7)	7105(6)	7746(5)	44(2)

C(62)	5144(7)	7760(6)	7302(5)	51(2)
C(63)	4226(8)	8016(8)	7724(7)	66(2)
C(64)	3868(9)	7638(9)	8548(7)	77(3)
C(65)	4388(10)	6996(10)	8970(7)	88(3)
C(66)	5313(8)	6739(8)	8574(6)	67(2)
C(71)	6641(7)	5436(6)	7554(5)	48(2)
C(72)	5782(8)	4604(7)	7013(6)	62(2)
C(73)	5381(10)	3546(8)	7254(7)	75(3)
C(74)	5898(12)	3302(9)	8052(8)	93(4)
C(75)	6777(12)	4095(10)	8572(7)	90(4)
C(76)	7151(9)	5162(8)	8350(6)	67(2)
C(81)	8305(7)	7677(6)	7901(4)	45(2)
C(82)	9357(8)	7428(8)	7953(5)	63(2)
C(83)	10418(9)	8111(11)	8469(6)	80(3)
C(84)	10436(9)	9058(11)	8885(6)	88(4)
C(85)	9420(10)	9332(8)	8810(5)	74(3)
C(86)	8342(8)	8649(6)	8321(5)	54(2)
O(20)	12496(6)	8263(5)	624(4)	72(2)

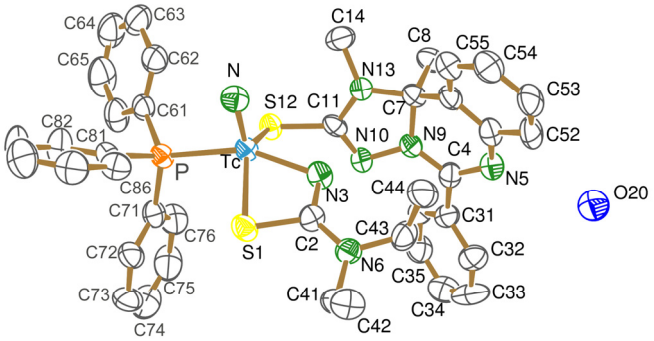

Figure 49 Ellipsoid plot (50% probability) of [TcN(PPh_3)(Et_2tu)(L^{7b})].

50 H_2L^{8a}

Table 50.1 Crystal data and structure refinement for H_2L^{8a}.

Empirical formula	$C_{26}H_{28}N_4OS$	
Formula weight	444.58	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.854(1) Å	α= 90°
	b = 7.782(1) Å	β= 99.77(1)°
	c = 28.473(2) Å	γ = 90°
Volume	2370.2(4) $Å^3$	
Z	4	
Density (calculated)	1.246 g/cm^3	
Absorption coefficient	0.162 mm^{-1}	
F(000)	944	
Crystal description	Plate	
Crystal color	Yellow	
Crystal size	0.2 x 0.2 x 0.05 mm^3	
Theta range for data collection	2.19 to 29.23	
Index ranges	-12<=h<=14, -10<=k<=10, -38<=l<=38	
Reflections collected	18087	
Independent reflections	6362 [R(int) = 0.0469]	
Completeness to theta = 29.23°	98.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6362 / 0 / 290	
Goodness-of-fit on F^2	1.085	
Final R indices [I>2sigma(I)]	R1 = 0.0467, wR2 = 0.1348	
R indices (all data)	R1 = 0.0601, wR2 = 0.1507	
Extinction coefficient	0.046(4)	
Largest diff. peak and hole	0.460 and -0.523 $e.Å^{-3}$	

Table 50.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for H_2L^{8a}. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	2375(1)	6648(1)	1887(1)	40(1)
C(2)	2606(1)	8017(2)	1451(1)	30(1)
N(3)	1684(1)	8329(1)	1067(1)	31(1)
C(4)	606(1)	8991(2)	1097(1)	29(1)
N(5)	-343(1)	8802(1)	726(1)	33(1)
C(7)	-156(1)	7906(2)	297(1)	30(1)
N(8)	-169(1)	6599(1)	-605(1)	31(1)
C(9)	31(1)	5745(2)	-972(1)	31(1)
C(31)	311(1)	10005(2)	1506(1)	30(1)
C(32)	1197(1)	11138(2)	1748(1)	36(1)
C(33)	915(2)	12124(2)	2122(1)	42(1)
C(34)	-234(2)	11980(2)	2263(1)	43(1)
C(35)	-1123(2)	10861(2)	2026(1)	40(1)
C(36)	-857(1)	9885(2)	1646(1)	35(1)
N(6)	3716(1)	8727(2)	1426(1)	37(1)
C(41)	3872(2)	9926(2)	1041(1)	47(1)
C(42)	4354(2)	9059(3)	633(1)	68(1)
C(43)	4815(2)	8385(3)	1785(1)	56(1)
C(44)	4927(3)	9618(4)	2200(1)	92(1)
C(51)	-1347(1)	7181(2)	19(1)	30(1)
C(52)	-2453(1)	7088(2)	201(1)	39(1)
C(53)	-3524(2)	6378(2)	-63(1)	45(1)
C(54)	-3502(1)	5748(2)	-513(1)	42(1)
C(55)	-2411(1)	5817(2)	-705(1)	37(1)
C(56)	-1328(1)	6517(2)	-440(1)	30(1)
C(61)	1226(1)	5828(2)	-1133(1)	32(1)
C(62)	2200(1)	6871(2)	-901(1)	37(1)
C(63)	3336(2)	6901(2)	-1066(1)	49(1)
C(64)	3500(2)	5926(3)	-1452(1)	55(1)
C(65)	2546(2)	4906(2)	-1688(1)	49(1)
C(66)	1419(1)	4862(2)	-1529(1)	39(1)
O(67)	2052(1)	7858(1)	-526(1)	45(1)

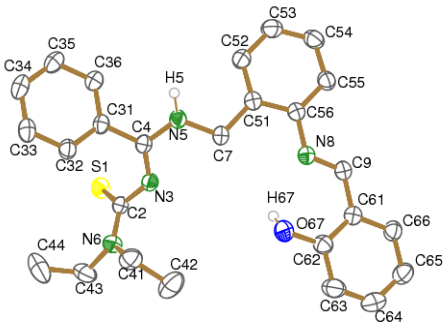

Figure 50 Ellipsoid plot (50% probability) of H_2L^{8a}.

51 $[ReOCl_2(HL^8)]$, (42)

Table 51.1 Crystal data and structure refinement for $[ReOCl_2(HL^8)] \cdot MeOH$.

Empirical formula	$C_{27}H_{31}Cl_2N_4O_3ReS$	
Formula weight	748.72	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 15.859(1) Å	α= 90°
	b = 16.517(2) Å	β= 90°
	c = 21.889(1) Å	γ = 90°
Volume	5733.8(8) $Å^3$	
Z	8	
Density (calculated)	1.735 g/cm^3	
Absorption coefficient	4.534 mm^{-1}	
F(000)	2960	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.200 x 0.133 x 0.050 mm^3	
Theta range for data collection	2.01 to 29.23	
Index ranges	-16<=h<=21, -16<=k<=22, -29<=l<=29	
Reflections collected	26695	
Independent reflections	7550 [R(int) = 0.2482]	
Completeness to theta = 29.23°	96.9 %	
Absorption correction	Integration	
Max. and min. transmission	0.8177 and 0.6028	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7550 / 0 / 345	
Goodness-of-fit on F^2	0.675	
Final R indices [I>2sigma(I)]	R1 = 0.0518, wR2 = 0.0592	
R indices (all data)	R1 = 0.1923, wR2 = 0.0878	
Largest diff. peak and hole	0.762 and -1.188 $e.Å^{-3}$	

Table 51.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[ReOCl_2(HL^8)] \cdot MeOH$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	2983(1)	2075(1)	9763(1)	21(1)
O(10)	3975(4)	1864(4)	9544(2)	26(2)
Cl(1)	2376(2)	2411(2)	8788(1)	36(1)
Cl(2)	2351(2)	793(2)	9616(1)	30(1)
S(1)	3086(2)	1578(2)	10793(1)	26(1)
C(2)	3518(6)	2274(6)	11315(4)	24(2)
N(3)	4189(5)	2728(5)	11169(3)	20(2)
C(4)	4851(6)	2429(6)	10897(4)	22(2)
N(5)	5314(4)	2907(8)	10549(3)	23(2)
C(7)	5067(7)	3741(6)	10396(4)	27(3)
N(8)	3388(5)	3329(5)	9862(3)	23(2)
C(9)	3047(7)	3856(5)	10191(4)	24(2)
C(31)	5168(6)	1590(7)	10993(4)	23(2)
C(32)	5428(7)	1106(8)	10510(5)	27(3)
C(33)	5736(7)	343(8)	10613(5)	35(3)
C(34)	5808(7)	68(8)	11207(5)	39(3)
C(35)	5530(8)	530(8)	11691(5)	39(3)
C(36)	5195(7)	1278(7)	11587(5)	33(3)
N(6)	3166(5)	2373(5)	11854(3)	35(2)
C(42)	2488(9)	1170(9)	12341(6)	56(4)
C(41)	2349(6)	1982(9)	12042(4)	40(3)
C(43)	3592(7)	2869(9)	12341(4)	41(3)
C(44)	3311(10)	3728(8)	12330(6)	63(5)
C(51)	4867(6)	3829(5)	9717(5)	22(2)
C(52)	5498(7)	4102(7)	9345(5)	29(3)
C(53)	5340(7)	4219(8)	8724(5)	39(3)
C(54)	4571(9)	4006(9)	8479(5)	52(4)
C(55)	3922(8)	3738(8)	8853(5)	48(4)
C(56)	4088(6)	3626(6)	9476(4)	20(2)
C(61)	2319(6)	3745(6)	10569(4)	25(2)
C(62)	2121(7)	4375(7)	10992(4)	37(3)
C(63)	1383(8)	4321(8)	11359(5)	49(4)
C(64)	868(8)	3617(8)	11311(5)	42(3)
C(65)	1061(6)	3048(8)	10892(5)	35(3)

C(66)	1768(6)	3105(6)	10507(4)	22(2)
O(67)	1891(4)	2549(4)	10074(2)	21(2)
O(71)	945(9)	3215(13)	12921(6)	135(7)
C(72)	840(20)	4062(15)	13030(10)	164(15)

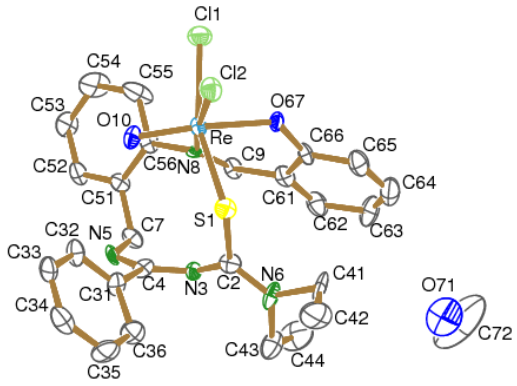

Figure 51 Ellipsoid plot (50% probability) of $[ReOCl_2(HL^8)] \cdot MeOH$.

52 [ReN(PPh$_3$)L^8], (43)

Table 52.1 Crystal data and structure refinement for [ReN(PPh$_3$)L^8] · Me$_2$CO.

Empirical formula	$C_{47}H_{45}N_5O_3PReS$	
Formula weight	977.16	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 10.452(1) Å	α= 99.23(1)°
	b = 13.134(1) Å	β= 98.45(1)°
	c = 15.752(1) Å	γ = 99.78(1)°
Volume	2069.2(3) Å^3	
Z	2	
Density (calculated)	1.568 g/cm^3	
Absorption coefficient	3.074 mm^{-1}	
F(000)	984	
Crystal description	Prism	
Crystal color	Red	
Crystal size	0.200 x 0.133 x 0.050 mm^3	
Theta range for data collection	1.88 to 29.26	
Index ranges	-12<=h<=14, -17<=k<=18, -21<=l<=20	
Reflections collected	21614	
Independent reflections	11037 [R(int) = 0.0902]	
Completeness to theta = 29.26°	97.8 %	
Absorption correction	Integration	
Max. and min. transmission	0.8267 and 0.5970	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11037 / 0 / 551	
Goodness-of-fit on F^2	0.810	
Final R indices [I>2sigma(I)]	R1 = 0.0429, wR2 = 0.0818	
R indices (all data)	R1 = 0.0905, wR2 = 0.1037	
Largest diff. peak and hole	0.894 and -1.615 e.Å^{-3}	

Table 52.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[ReN(PPh_3)L^8] \cdot Me_2CO$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	2240(1)	2222(1)	2170(1)	26(1)
N(10)	1787(6)	3371(4)	2134(3)	37(1)
S(1)	4571(2)	2709(2)	2655(1)	41(1)
C(2)	5060(7)	3296(5)	1810(4)	35(2)
N(3)	4689(5)	2902(4)	970(3)	35(1)
C(4)	3670(6)	2127(4)	563(4)	28(1)
N(5)	2561(5)	1851(4)	854(3)	30(1)
C(31)	3932(6)	1601(5)	-290(4)	31(1)
C(32)	3802(7)	516(5)	-484(4)	37(2)
C(33)	4215(8)	58(7)	-1227(5)	52(2)
C(34)	4717(8)	656(8)	-1777(5)	61(2)
C(35)	4808(8)	1728(7)	-1603(4)	58(2)
C(36)	4432(7)	2209(6)	-854(4)	44(2)
N(6)	5975(7)	4208(5)	2024(4)	56(2)
C(41)	6469(9)	4779(7)	2924(6)	74(3)
C(42)	7928(11)	5199(10)	3019(9)	112(5)
O(43)	8206(8)	5852(6)	2450(7)	118(3)
C(44)	7770(10)	5282(8)	1571(8)	99(4)
C(45)	6324(10)	4819(7)	1381(7)	73(3)
C(7)	1427(6)	1230(5)	194(4)	32(1)
C(51)	195(6)	1675(5)	225(4)	32(1)
C(52)	-460(7)	1992(5)	-500(4)	38(2)
C(53)	-1662(8)	2306(6)	-475(5)	49(2)
C(54)	-2206(7)	2334(6)	268(5)	45(2)
C(55)	-1571(7)	2038(5)	993(4)	39(2)
C(56)	-392(6)	1711(4)	963(4)	30(1)
N(8)	292(5)	1326(4)	1678(3)	30(1)
C(9)	-304(6)	476(5)	1884(4)	32(1)
C(61)	283(7)	-140(5)	2425(4)	34(1)
C(62)	-559(8)	-981(5)	2663(4)	48(2)
C(63)	-55(11)	-1670(6)	3099(5)	62(2)
C(64)	1285(11)	-1590(6)	3319(5)	60(2)
C(65)	2132(9)	-818(6)	3079(5)	50(2)
C(66)	1677(7)	-54(5)	2630(4)	33(1)

O(67)	2503(4)	658(3)	2397(3)	34(1)
P	1879(2)	2548(1)	3686(1)	29(1)
C(71)	2883(7)	2122(5)	4564(4)	36(2)
C(72)	2717(8)	2411(6)	5430(4)	48(2)
C(73)	3515(8)	2166(7)	6103(4)	55(2)
C(74)	4508(8)	1635(6)	5943(4)	52(2)
C(75)	4685(7)	1352(6)	5106(4)	44(2)
C(76)	3868(7)	1569(5)	4410(4)	38(2)
C(81)	175(7)	2072(5)	3807(4)	36(2)
C(82)	-193(8)	1305(6)	4299(5)	52(2)
C(83)	-1500(9)	925(7)	4287(6)	62(2)
C(84)	-2461(9)	1263(7)	3795(6)	62(2)
C(85)	-2128(8)	2015(7)	3321(5)	56(2)
C(86)	-817(7)	2424(6)	3319(4)	44(2)
C(91)	2203(7)	3981(5)	4115(4)	33(1)
C(92)	1300(11)	4497(9)	4425(10)	150(7)
C(93)	1627(13)	5605(9)	4704(12)	169(9)
C(94)	2798(10)	6135(7)	4737(6)	67(3)
C(95)	3671(10)	5640(6)	4447(7)	75(3)
C(96)	3354(10)	4570(6)	4109(7)	80(3)
C(100)	2548(14)	5539(9)	1553(13)	97(4)
C(11)	1460(30)	5760(20)	2100(30)	100(11)
C(12)	2320(30)	4929(16)	803(12)	79(9)
O(10)	3708(18)	5882(13)	1999(16)	111(8)
C(11A)	3310(30)	5620(20)	987(19)	75(9)
C(12A)	1120(30)	4950(30)	800(20)	115(13)
O(10A)	2360(70)	6050(30)	2230(20)	250(40)

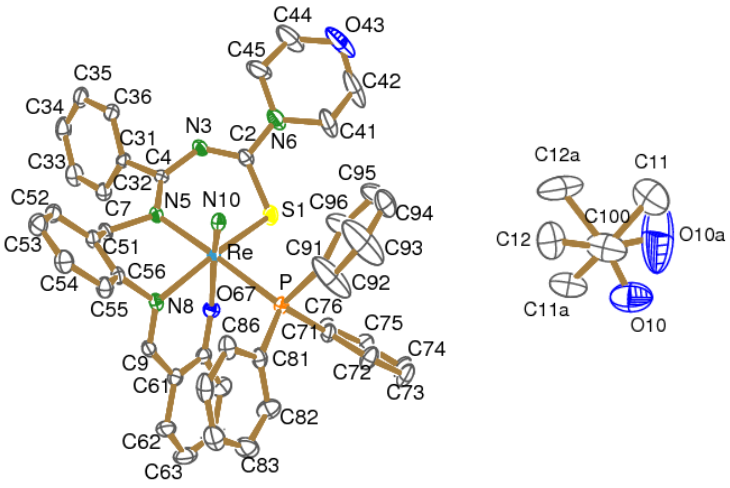

Figure 52 Ellipsoid plot (50% probability) of $[ReN(PPh_3)L^8] \cdot Me_2CO$

53 [ReO(L^9)], (44)

Table 53.1 Crystal data and structure refinement for [ReO(L^9)] · H_2O.

Empirical formula	$C_{20}H_{26}N_5O_6ReS$	
Formula weight	650.72	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 11.290(1) Å	α= 93.48(1)°
	b = 11.310(1) Å	β= 110.42(1)°
	c = 12.241(1) Å	γ = 118.26(1)°
Volume	1240.95(19) $Å^3$	
Z	2	
Density (calculated)	1.741 g/cm^3	
Absorption coefficient	5.024 mm^{-1}	
F(000)	640	
Crystal description	Block	
Crystal color	Red-brown	
Crystal size	0.210 x 0.193 x 0.180 mm^3	
Theta range for data collection	1.85 to 29.23	
Index ranges	-15<=h<=15, -15<=k<=15, -16<=l<=16	
Reflections collected	23724	
Independent reflections	6706 [R(int) = 0.0689]	
Completeness to theta = 29.23°	99.4 %	
Absorption correction	Integration	
Max. and min. transmission	0.5594 and 0.3129	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6706 / 0 / 336	
Goodness-of-fit on F^2	1.182	
Final R indices [I>2sigma(I)]	R1 = 0.0332, wR2 = 0.0827	
R indices (all data)	R1 = 0.0408, wR2 = 0.0988	
Extinction coefficient	0.0276(12)	
Largest diff. peak and hole	2.056 and -1.154 e.$Å^{-3}$	

Table 53.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[ReO(L^9)] \cdot H_2O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	5512(1)	6534(1)	6882(1)	27(1)
O(10)	6298(4)	6388(4)	5989(3)	39(1)
S(1)	6406(1)	8876(1)	7532(1)	37(1)
C(2)	4957(6)	9135(5)	6748(4)	37(1)
N(3)	3842(5)	8341(4)	5654(4)	33(1)
C(4)	3208(5)	6991(5)	5216(4)	29(1)
N(5)	3483(4)	6115(4)	5808(3)	29(1)
C(7)	2257(5)	4651(5)	5482(4)	33(1)
C(8)	2752(5)	3943(5)	6382(4)	31(1)
O(9)	1887(5)	2813(4)	6447(4)	44(1)
N(10)	4243(5)	4707(4)	7082(3)	31(1)
C(11)	4847(6)	4284(5)	8137(4)	38(1)
C(12)	6338(6)	5534(6)	9003(4)	36(1)
O(13)	7053(5)	5476(5)	10000(3)	47(1)
N(14)	6796(5)	6686(4)	8604(3)	33(1)
C(15)	8179(6)	7912(6)	9484(4)	36(1)
C(16)	8012(7)	8533(6)	10504(5)	47(1)
O(17)	6874(6)	8258(5)	10547(4)	53(1)
O(18A)	9444(16)	9271(13)	11447(8)	52(3)
C(19A)	9630(20)	9904(18)	12592(13)	76(7)
C(20A)	9690(30)	11110(30)	12500(30)	109(11)
O(18B)	9218(14)	9740(20)	11191(16)	58(5)
C(19B)	9010(50)	10170(50)	12300(30)	140(20)
C(20B)	10060(30)	10520(30)	13315(17)	96(9)
O(21)	6172(7)	3424(6)	11263(5)	26(1)
O(22)	8297(9)	3029(9)	12998(7)	42(2)
C(31)	2063(5)	6428(5)	3938(4)	30(1)
C(32)	1065(6)	6865(6)	3557(4)	37(1)
C(33)	20(6)	6360(7)	2356(5)	43(1)
C(34)	-12(7)	5441(7)	1529(5)	45(1)
C(35)	1005(7)	5017(7)	1902(4)	43(1)
C(36)	2039(6)	5505(6)	3104(4)	35(1)
N(6)	4992(7)	10223(6)	7224(5)	56(1)
C(41)	3707(7)	10396(6)	6738(5)	43(1)

C(42)	6381(10)	11392(8)	8290(7)	66(2)
C(43)	6242(16)	11131(13)	9413(9)	99(4)
C(44)	3801(9)	11180(8)	5804(6)	57(2)

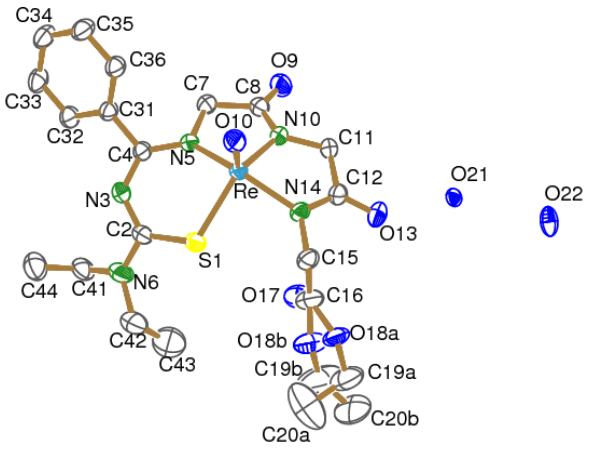

Figure 53 Ellipsoid plot (50% probability) of $[ReO(L^9)] \cdot H_2O$.

54 [ReOL^{10}], (45)

Table 54.1 Crystal data and structure refinement for [ReOL^{10}].

Empirical formula	$C_{23}H_{25}N_4O_5ReS$	
Formula weight	655.73	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 32.448(3) Å	α= 90°
	b = 7.160(1) Å	β= 100.69(1)°
	c = 20.521(2) Å	γ = 90°
Volume	4684.8(9) Å^3	
Z	8	
Density (calculated)	1.859 g/cm^3	
Absorption coefficient	5.320 mm^{-1}	
F(000)	2576	
Crystal description	Needle	
Crystal color	Violet	
Crystal size	0.500 x 0.203 x 0.040 mm^3	
Theta range for data collection	2.56 to 29.41	
Index ranges	-44<=h<=44, -8<=k<=9, -20<=l<=28	
Reflections collected	14462	
Independent reflections	6207 [R(int) = 0.1002]	
Completeness to theta = 29.41°	96.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.7838 and 0.5209	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6207 / 0 / 326	
Goodness-of-fit on F^2	0.872	
Final R indices [I>2sigma(I)]	R1 = 0.0477, wR2 = 0.0776	
R indices (all data)	R1 = 0.1250, wR2 = 0.0946	
Extinction coefficient	0.00097(6)	
Largest diff. peak and hole	1.215 and -2.495 e.Å^{-3}	

Table 54.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$ReOL^{10}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	8660(1)	1231(1)	9065(1)	31(1)
O(10)	8372(1)	1993(7)	8363(3)	39(1)
S(1)	9303(1)	2572(3)	9088(1)	42(1)
C(2)	9598(2)	946(14)	8729(5)	49(2)
N(3)	9445(2)	-337(10)	8291(4)	41(2)
C(4)	9093(2)	-1313(13)	8273(4)	35(2)
N(5)	8837(2)	-1224(10)	8719(3)	32(1)
C(31)	8972(2)	-2538(13)	7688(5)	41(2)
C(32)	9168(3)	-4238(14)	7633(6)	65(3)
C(33)	9020(4)	-5405(17)	7089(8)	85(4)
C(34)	8710(4)	-4820(20)	6604(7)	75(4)
C(35)	8531(3)	-3162(19)	6639(6)	71(3)
C(36)	8646(3)	-1989(16)	7183(6)	61(3)
N(6)	10014(2)	1152(15)	8856(5)	73(3)
C(41)	10285(3)	-54(17)	8520(6)	66(3)
C(42)	10349(3)	-1902(18)	8834(7)	77(4)
C(43)	10213(6)	2900(50)	9145(12)	44(5)
C(44)	10330(9)	2570(40)	9914(13)	66(8)
C(43A)	10227(6)	2070(40)	9550(30)	78(9)
C(44A)	10376(12)	3610(60)	9380(30)	117(13)
C(7)	8574(2)	-2859(11)	8814(5)	32(2)
C(51)	8102(2)	-2619(11)	8561(4)	32(2)
C(52)	7900(2)	-3886(13)	8106(4)	38(2)
C(53)	7461(2)	-3944(13)	7916(4)	41(2)
C(54)	7229(2)	-2638(11)	8188(5)	38(2)
C(55)	7422(2)	-1327(13)	8630(4)	36(2)
C(56)	7857(2)	-1288(12)	8805(4)	31(2)
N(9)	8061(2)	60(9)	9305(3)	29(1)
C(61)	7819(2)	1840(10)	9309(4)	33(2)
C(62)	8084(2)	3398(10)	9668(4)	32(2)
O(63)	7924(2)	4638(8)	9942(3)	46(2)
O(64)	8481(1)	3408(7)	9620(3)	39(1)
C(65)	8134(2)	-810(10)	9982(4)	32(2)
C(66)	8583(2)	-574(11)	10347(5)	38(2)

O(67)	8684(2)	-1039(11)	10916(3)	55(2)
O(68)	8835(1)	93(8)	9976(3)	41(1)

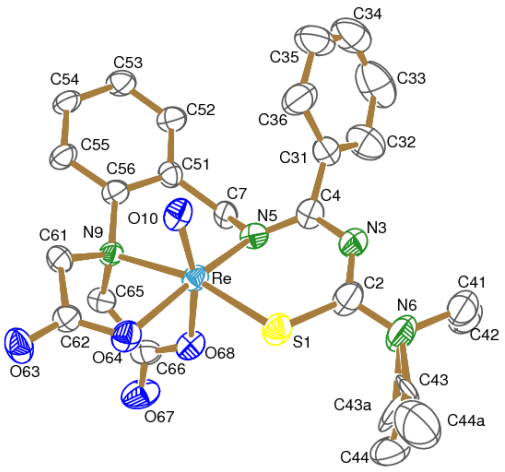

Figure 54 Ellipsoid plot (50% probability) of [ReOL^{10}].

55 [TcOL^{10}], (46)

Table 55.1 Crystal data and structure refinement for [TcOL^{10}].

Empirical formula	$C_{23}H_{25}N_4O_5STc$	
Formula weight	567.53	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 32.213(2) Å	α= 90°
	b = 7.187(1) Å	β= 100.29(1)°
	c = 20.453(2) Å	γ = 90°
Volume	4659.2(8) Å^3	
Z	8	
Density (calculated)	1.618 g/cm^3	
Absorption coefficient	0.751 mm^{-1}	
F(000)	2320	
Crystal description	Plate	
Crystal color	Green	
Crystal size	0.20 x 0.15 x 0.03 mm^3	
Theta range for data collection	2.02 to 29.22	
Index ranges	-44<=h<=44, -9<=k<=9, -28<=l<=25	
Reflections collected	20479	
Independent reflections	6270 [R(int) = 0.1119]	
Completeness to theta = 29.22°	99.3 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6270 / 0 / 326	
Goodness-of-fit on F^2	0.907	
Final R indices [I>2sigma(I)]	R1 = 0.0505, wR2 = 0.1109	
R indices (all data)	R1 = 0.0963, wR2 = 0.1355	
Extinction coefficient	0.00176(19)	
Largest diff. peak and hole	2.561 and -1.025 e.Å^{-3}	

Table 55.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)for [$TcOL^{10}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	6344(1)	8779(1)	5936(1)	26(1)
O(10)	6629(1)	8033(4)	6643(2)	34(1)
S(1)	5694(1)	7440(2)	5905(1)	37(1)
C(2)	5394(2)	9008(7)	6278(3)	40(1)
N(3)	5550(1)	10320(6)	6721(2)	34(1)
C(4)	5900(1)	11280(7)	6730(2)	29(1)
N(5)	6162(1)	11189(5)	6284(2)	27(1)
C(31)	6009(2)	12572(7)	7302(2)	36(1)
C(32)	6332(2)	12064(9)	7818(3)	48(1)
C(34)	6274(2)	14888(12)	8376(4)	73(2)
C(35)	5958(3)	15454(9)	7883(5)	80(2)
C(33)	6452(2)	13221(12)	8347(3)	70(2)
C(36)	5810(2)	14251(8)	7336(3)	61(2)
N(6)	4978(1)	8813(8)	6165(3)	65(2)
C(41)	4703(2)	10022(10)	6484(3)	57(2)
C(42)	4638(2)	11889(11)	6174(3)	65(2)
C(43A)	4789(3)	7057(18)	5844(6)	37(2)
C(44A)	4657(4)	7419(18)	5086(6)	52(4)
C(43B)	4754(4)	8010(20)	5444(10)	63(4)
C(44B)	4636(6)	6270(30)	5654(11)	96(6)
C(7)	6428(1)	12831(6)	6186(2)	26(1)
C(51)	6899(1)	12613(6)	6432(2)	24(1)
C(52)	7101(1)	13900(6)	6897(2)	31(1)
C(53)	7538(1)	13919(7)	7083(2)	36(1)
C(54)	7776(1)	12661(7)	6821(2)	34(1)
C(55)	7587(1)	11326(7)	6367(2)	31(1)
C(56)	7147(1)	11310(6)	6188(2)	26(1)
N(9)	6946(1)	9953(5)	5688(2)	25(1)
C(61)	7186(1)	8177(6)	5687(3)	33(1)
C(62)	6909(2)	6593(6)	5336(2)	34(1)
O(63)	7075(1)	5373(5)	5057(2)	46(1)
O(64)	6517(1)	6594(4)	5389(2)	39(1)
C(65)	6878(1)	10822(6)	5009(2)	31(1)
C(66)	6427(2)	10559(6)	4652(2)	33(1)

O(67)	6330(1)	10990(6)	4068(2)	49(1)
O(68)	6172(1)	9924(5)	5020(2)	32(1)

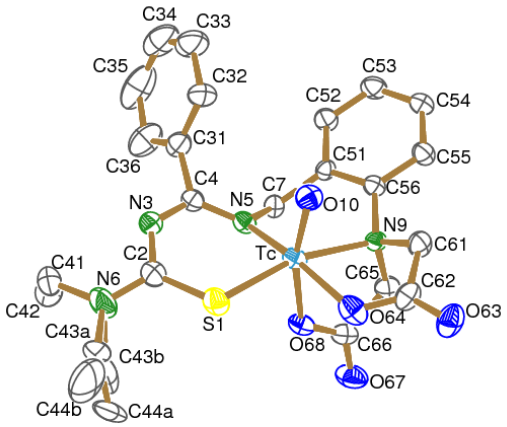

Figure 55 Ellipsoid plot (50% probability) of [TcOL^{10}].

56 [ReOL^{11}], (47)

Table 56.1 Crystal data and structure refinement for [ReOL^{11}].

Empirical formula	$C_{23}H_{32}N_4O_6ReS$	
Formula weight	678.79	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.562 (1) Å	α= 90°
	b = 22.684(1) Å	β= 94.24(1)°
	c = 10.621(1) Å	γ = 90°
Volume	2297.4(3) Å^3	
Z	4	
Density (calculated)	1.962 g/cm^3	
Absorption coefficient	5.430 mm^{-1}	
F(000)	1348	
Crystal description	Plate	
Crystal color	Red	
Crystal size	0.110 x 0.083 x 0.030 mm^3	
Theta range for data collection	1.80 to 26.90	
Index ranges	-12<=h<=8, -28<=k<=28, -13<=l<=13	
Reflections collected	13961	
Independent reflections	4871 [R(int) = 0.0936]	
Completeness to theta = 26.90°	97.9 %	
Absorption correction	Integration	
Max. and min. transmission	0.8103 and 0.5618	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4871 / 0 / 317	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0463, wR2 = 0.0882	
R indices (all data)	R1 = 0.0883, wR2 = 0.1191	
Extinction coefficient	0.0035(3)	
Largest diff. peak and hole	1.127 and -0.821 e.Å^{-3}	

Table 56.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$ReOL^{11}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	9410(1)	8361(1)	8137(1)	21(1)
O(10)	10175(8)	8734(3)	7016(6)	27(2)
S(1)	10492(3)	8730(1)	9988(2)	29(1)
C(2)	9275(12)	9236(4)	10523(9)	27(2)
N(3)	8478(9)	9589(4)	9743(7)	26(2)
C(4)	7768(10)	9442(4)	8679(9)	20(2)
N(5)	7704(9)	8898(4)	8164(7)	24(2)
C(31)	6989(10)	9941(4)	8020(9)	24(2)
C(32)	6418(12)	10378(4)	8751(10)	27(2)
C(33)	5655(13)	10830(5)	8167(11)	35(3)
C(34)	5482(13)	10871(4)	6870(11)	32(2)
C(35)	6075(12)	10442(5)	6165(10)	33(2)
C(36)	6793(11)	9974(4)	6720(9)	26(2)
N(6)	9122(10)	9292(4)	11758(8)	34(2)
C(41)	7934(16)	9619(7)	12218(12)	55(4)
C(42)	8295(15)	9884(6)	13461(11)	47(3)
O(43)	8801(9)	9455(3)	14341(7)	40(2)
C(44)	10064(14)	9215(6)	13942(11)	42(3)
C(45)	9829(15)	8908(5)	12718(10)	41(3)
C(51)	6370(10)	8720(4)	7601(9)	22(2)
C(52)	5189(11)	8855(5)	8213(10)	30(2)
C(53)	3871(12)	8695(5)	7740(11)	36(3)
C(54)	3705(11)	8372(6)	6602(10)	37(2)
C(55)	4909(12)	8221(5)	6013(9)	32(2)
C(56)	6234(10)	8381(5)	6478(9)	27(2)
C(7)	7450(12)	8185(4)	5769(9)	28(2)
N(8)	8408(9)	7797(3)	6637(7)	21(2)
C(61)	9664(12)	7545(5)	6062(11)	33(3)
C(62)	10755(12)	7345(4)	7104(10)	29(2)
O(63)	11492(9)	6927(3)	6960(8)	40(2)
O(64)	10779(8)	7662(3)	8150(7)	30(2)
C(65)	7617(11)	7298(4)	7166(9)	26(2)
C(66)	7457(11)	7379(5)	8563(10)	27(2)
O(67)	6717(9)	7039(4)	9111(7)	40(2)

O(68)	8214(7)	7794(3)	9089(6)	26(2)

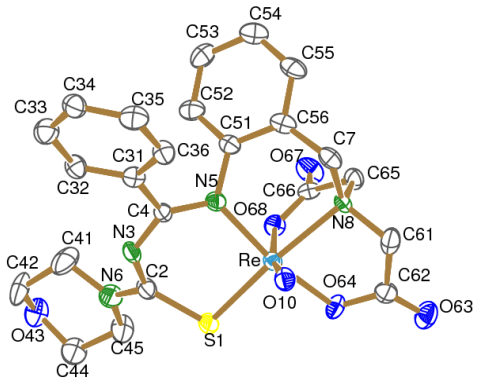

Figure 56 Ellipsoid plot (50% probability) of [ReOL[11]].

57 [Re(NPh)L^{11a}], (48a)

Table 57.1 Crystal data and structure refinement for [Re(NPh)L^{11a}].

Empirical formula	$C_{29}H_{28}N_5O_5ReS$	
Formula weight	744.82	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P $2_12_12_1$	
Unit cell dimensions	a = 7.812(1) Å	α= 90°
	b = 8.564(1) Å	β= 90°
	c = 40.864(3) Å	γ = 90°
Volume	2734.3(4) Å^3	
Z	4	
Density (calculated)	1.809 g/cm^3	
Absorption coefficient	4.571 mm^{-1}	
F(000)	1472	
Crystal description	Block	
Crystal color	Green	
Crystal size	0.100 x 0.067 x 0.050 mm^3	
Theta range for data collection	2.58 to 29.30	
Index ranges	-6<=h<=10, -11<=k<=10, -55<=l<=56	
Reflections collected	14880	
Independent reflections	6831 [R(int) = 0.0997]	
Completeness to theta = 29.30°	97.2 %	
Absorption correction	Integration	
Max. and min. transmission	0.7348 and 0.5504	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6831 / 0 / 371	
Goodness-of-fit on F^2	0.882	
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.1122	
R indices (all data)	R1 = 0.1118, wR2 = 0.1399	
Absolute structure parameter	-0.030(18)	
Extinction coefficient	0.0120(6)	
Largest diff. peak and hole	1.591 and -1.856 e.Å^{-3}	

Table 57.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Re(NPh)L^{11a}]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	9179(1)	8638(1)	8466(1)	38(1)
S(1)	10523(5)	10715(4)	8734(1)	46(1)
C(2)	9974(16)	10548(14)	9142(3)	36(3)
N(3)	8551(14)	9886(11)	9257(2)	38(2)
C(4)	7710(15)	8673(15)	9135(2)	35(2)
N(5)	8189(13)	7726(12)	8890(2)	40(2)
C(31)	6015(16)	8439(13)	9298(2)	37(2)
C(32)	5814(18)	8642(15)	9638(2)	42(2)
C(33)	4300(20)	8332(14)	9778(2)	48(3)
C(34)	2895(19)	7904(15)	9603(3)	47(3)
C(35)	3041(17)	7745(15)	9256(3)	44(3)
C(36)	4580(15)	8024(15)	9117(3)	42(3)
N(6)	10950(14)	11310(13)	9362(2)	46(2)
C(41)	10616(19)	11258(14)	9716(2)	45(3)
C(42)	10690(20)	12913(16)	9859(3)	51(3)
O(43)	12361(13)	13650(13)	9783(2)	58(2)
C(44)	12610(20)	13711(18)	9442(3)	61(4)
C(45)	12580(20)	12108(16)	9284(3)	52(3)
C(51)	7673(17)	6113(14)	8913(2)	38(3)
C(52)	7844(18)	5378(15)	9217(3)	44(3)
C(53)	7278(18)	3859(17)	9260(3)	48(3)
C(54)	6616(19)	3025(17)	9001(3)	54(4)
C(55)	6509(17)	3738(17)	8699(3)	48(3)
C(56)	7065(16)	5297(15)	8648(2)	37(3)
C(7)	6883(19)	5922(16)	8312(2)	45(3)
N(8)	8616(12)	6539(12)	8193(2)	38(2)
C(61)	8484(18)	7096(16)	7846(2)	46(3)
C(62)	9810(17)	8314(15)	7771(3)	45(3)
O(63)	10157(12)	8613(12)	7485(2)	53(2)
O(64)	10438(11)	9030(10)	8022(2)	46(2)
C(65)	10012(18)	5365(16)	8233(3)	49(3)
C(66)	11528(19)	6015(18)	8405(3)	59(4)
O(67)	12868(19)	5284(19)	8416(4)	143(7)
O(68)	11299(13)	7322(11)	8547(2)	54(3)

N(10)	7477(15)	9635(11)	8319(2)	40(2)
C(71)	6314(16)	10566(14)	8141(3)	39(3)
C(72)	7003(18)	11543(17)	7903(2)	52(3)
C(73)	5960(20)	12609(16)	7743(3)	55(3)
C(74)	4230(20)	12653(17)	7808(3)	59(4)
C(75)	3551(19)	11570(19)	8032(3)	59(4)
C(76)	4537(18)	10561(17)	8199(3)	50(4)

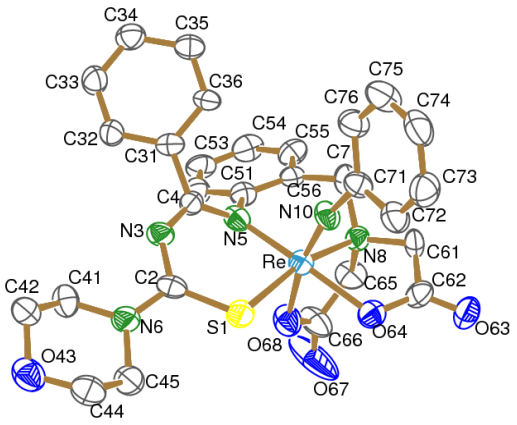

Figure 57 Ellipsoid plot (50% probability) of [Re(NPh)L[11a]].

58 [Re(NPh)L^{11b}], (48b)

Table 58.1 Crystal data and structure refinement for [Re(NPh)L^{11b}] · 1/2CH_2Cl_2.

Empirical formula	$C_{32.50}H_{29}ClN_5O_4ReS$	
Formula weight	807.32	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 27.316(2) Å	α= 90°
	b = 12.410(1) Å	β= 99.85(1)°
	c = 21.298(2) Å	γ = 90°
Volume	7113.0(1) Å^3	
Z	8	
Density (calculated)	1.508 g/cm^3	
Absorption coefficient	3.591 mm^{-1}	
F(000)	3192	
Crystal description	Plate	
Crystal color	Yellow-Green	
Crystal size	0.400 x 0.217 x 0.050 mm^3	
Theta range for data collection	1.51 to 26.87	
Index ranges	-32<=h<=34, -15<=k<=15, -26<=l<=26	
Reflections collected	21328	
Independent reflections	7541 [R(int) = 0.1547]	
Completeness to theta = 26.87°	98.5 %	
Absorption correction	Integration	
Max. and min. transmission	0.7903 and 0.5905	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7541 / 0 / 417	
Goodness-of-fit on F^2	1.114	
Final R indices [I>2sigma(I)]	R1 = 0.0835, wR2 = 0.1803	
R indices (all data)	R1 = 0.1555, wR2 = 0.2156	
Extinction coefficient	0.00214(14)	
Largest diff. peak and hole	3.496 and -1.230 e.Å^{-3}	

Table 58.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Re(NPh)L^{11b}] · 1/2CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	2962(1)	6604(1)	8257(1)	27(1)
S(1)	3174(1)	4959(3)	8762(2)	34(1)
C(2)	3810(6)	4952(13)	8915(7)	34(3)
N(3)	4106(5)	5790(10)	8994(6)	32(3)
C(4)	4064(5)	6750(12)	8698(8)	32(3)
N(5)	3688(4)	7015(10)	8227(6)	26(2)
C(31)	4457(6)	7548(13)	8967(8)	34(3)
C(32)	4925(6)	7183(14)	9209(8)	43(4)
C(33)	5301(7)	7940(20)	9453(10)	63(6)
C(34)	5183(8)	9009(18)	9448(10)	57(6)
C(35)	4726(7)	9350(16)	9210(9)	49(5)
C(36)	4359(7)	8629(14)	8970(8)	41(4)
N(6)	4054(5)	3976(11)	9008(7)	41(3)
C(41)	3804(6)	2975(14)	9037(9)	41(4)
C(42)	3522(7)	2799(14)	9517(9)	45(4)
C(43)	3305(7)	1755(17)	9572(10)	54(5)
C(44)	3369(8)	988(15)	9136(12)	61(6)
C(45)	3634(7)	1196(16)	8653(10)	52(5)
C(46)	3840(6)	2189(14)	8597(9)	40(4)
C(47)	4594(6)	3937(16)	9113(11)	54(6)
C(51)	3811(5)	7723(11)	7748(7)	24(3)
C(52)	4234(6)	7544(14)	7496(8)	42(4)
C(53)	4403(8)	8196(18)	7043(11)	65(7)
C(54)	4100(8)	9030(19)	6816(12)	67(7)
C(55)	3665(7)	9231(16)	7031(10)	51(5)
C(56)	3502(6)	8573(13)	7492(7)	33(3)
C(7)	3013(6)	8793(12)	7670(7)	31(3)
N(8)	2676(4)	7860(9)	7585(5)	24(2)
C(61)	2174(5)	8104(12)	7731(7)	32(3)
C(62)	1918(5)	7087(13)	7921(8)	34(4)
O(63)	1483(5)	7091(11)	7952(6)	49(3)
O(64)	2208(4)	6248(8)	8048(5)	34(2)
C(65)	2620(6)	7470(14)	6905(7)	37(4)
C(66)	2863(6)	6352(15)	6867(7)	42(5)

O(67)	2915(6)	6008(13)	6343(6)	66(4)
O(68)	2995(4)	5862(9)	7406(5)	38(3)
N(10)	2920(5)	7369(10)	8923(6)	30(3)
C(71)	2924(7)	8046(13)	9439(7)	40(4)
C(72)	3329(8)	8063(15)	9919(9)	50(5)
C(73)	3317(9)	8770(20)	10446(11)	70(7)
C(74)	2917(9)	9393(17)	10471(10)	60(6)
C(75)	2530(9)	9356(15)	10002(9)	56(5)
C(76)	2516(7)	8692(13)	9478(8)	39(4)
C(81)	3894(11)	4430(20)	6490(18)	40(8)
Cl(82)	4424(4)	4685(10)	7042(6)	76(4)
Cl(83)	4035(4)	4549(9)	5694(5)	60(3)

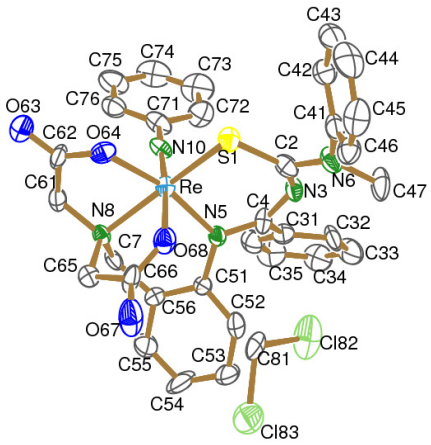

Figure 58 Ellipsoid plot (50% probability) of [Re(NPh)L^{11b}] · ½CH_2Cl_2.

59 [ReO(L^{10}-COOEt)], (49)

Table 59.1 Crystal data and structure refinement for [ReO(L^{10}-COOEt)] · 1/2MeOH.

Empirical formula	$C_{29.50}H_{29}N_4O_{7.50}ReS$	
Formula weight	777.83	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.949(1) Å	α= 100.54(1)°
	b = 10.980(1) Å	β= 92.32(1)°
	c = 16.796(2) Å	γ = 113.36(1)°
Volume	1643.0(3) $Å^3$	
Z	2	
Density (calculated)	1.572 g/cm^3	
Absorption coefficient	3.811 mm^{-1}	
F(000)	770	
Crystal description	Block	
Crystal color	Green	
Crystal size	0.100 x 0.067 x 0.050 mm^3	
Theta range for data collection	2.07 to 29.29	
Index ranges	-13<=h<=13, -15<=k<=15, -23<=l<=22	
Reflections collected	17975	
Independent reflections	8768 [R(int) = 0.1067]	
Completeness to theta = 29.29°	97.9 %	
Absorption correction	Integration	
Max. and min. transmission	0.6384 and 0.3695	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8768 / 0 / 384	
Goodness-of-fit on F^2	1.058	
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1750	
R indices (all data)	R1 = 0.0960, wR2 = 0.2006	
Extinction coefficient	0.038(2)	
Largest diff. peak and hole	2.662 and -2.230 e.$Å^{-3}$	

Table 59.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [ReO(L^{10}-COOEt)] · 1/2MeOH. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Re	6315(1)	8317(1)	7347(1)	41(1)
O(10)	7630(8)	9390(7)	6936(5)	57(2)
S(1)	5165(3)	9576(2)	8012(2)	52(1)
C(2)	6322(9)	10444(8)	8943(6)	44(2)
N(3)	7032(8)	9947(7)	9372(5)	45(2)
C(4)	7414(8)	8915(7)	9122(6)	39(2)
N(5)	7608(7)	8457(6)	8369(5)	38(1)
C(31)	7679(9)	8331(8)	9829(6)	40(2)
C(32)	6917(9)	6947(8)	9803(7)	46(2)
C(33)	7086(10)	6453(8)	10493(7)	48(2)
C(34)	8001(11)	7308(11)	11183(7)	55(2)
C(35)	8742(11)	8706(10)	11217(7)	53(2)
C(36)	8552(9)	9184(8)	10533(6)	46(2)
N(6)	6427(7)	11707(7)	9240(5)	44(2)
C(41)	7452(9)	12556(8)	9950(6)	42(2)
C(42)	6940(10)	13025(9)	10639(7)	49(2)
C(43)	7919(10)	13956(10)	11283(8)	54(2)
C(44)	9421(10)	14406(9)	11251(7)	48(2)
C(45)	9941(10)	13910(9)	10551(6)	47(2)
C(46)	8958(10)	12978(9)	9892(6)	45(2)
C(47)	5527(10)	12285(9)	8872(7)	52(2)
C(48)	10550(12)	15420(10)	11922(7)	55(2)
O(481)	11861(8)	15674(8)	11976(6)	71(2)
O(482)	9937(12)	15946(12)	12438(7)	98(4)
C(49)	11070(30)	17110(30)	13129(16)	171(15)
C(50)	10470(80)	16750(40)	13810(20)	410(50)
C(7)	8391(9)	7553(8)	8266(6)	41(2)
C(51)	9119(8)	7463(8)	7508(6)	42(2)
C(52)	10628(9)	7762(9)	7597(7)	49(2)
C(53)	11341(10)	7528(10)	6916(7)	54(2)
C(54)	10548(10)	6996(10)	6143(7)	54(2)
C(55)	9061(10)	6737(10)	6045(6)	49(2)
C(56)	8338(9)	6965(8)	6708(6)	43(2)
N(8)	6772(7)	6656(7)	6617(5)	39(1)

C(61)	6196(10)	6642(10)	5775(6)	47(2)
C(62)	4743(11)	6827(11)	5778(7)	57(2)
O(63)	3826(9)	6320(9)	5177(6)	76(3)
O(64)	4650(7)	7639(7)	6395(4)	54(2)
C(65)	5912(10)	5308(9)	6827(7)	52(2)
C(66)	4921(10)	5413(9)	7473(7)	53(2)
O(67)	4225(12)	4443(9)	7750(8)	97(4)
O(68)	4931(7)	6598(6)	7710(5)	48(2)
O(74)	7965	8085	3631	32(4)
C(73)	6880	8592	4136	54(9)
O(71)	7092	9455	4241	59(7)
C(72)	7278	9023	3238	53(8)

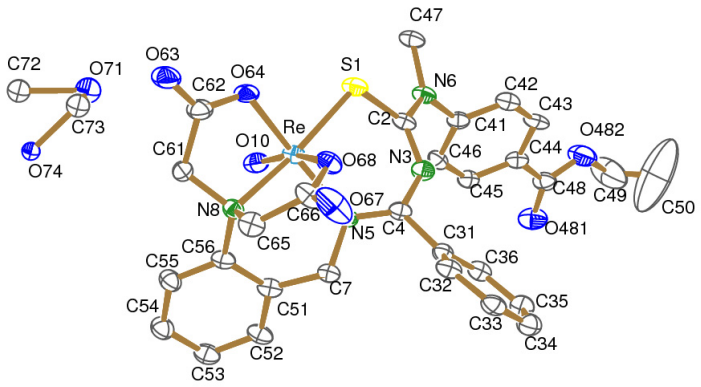

Figure 59 Ellipsoid plot (50% probability) of [ReO(L^{10}-COOEt)] · 1/2MeOH.

60 [TcO(L^{10}-COOEt)], (50)

Table 60.1 Crystal data and structure refinement for [TcO(L^{10}-COOEt)].

Empirical formula	$C_{29}H_{27}N_4O_7STc$	
Formula weight	673.61	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.993(1) Å	α= 100.30(1)°
	b = 11.505(2) Å	β= 89.20(1)°
	c = 16.440(2) Å	γ = 118.66(1)°
Volume	1626.2(4) $Å^3$	
Z	2	
Density (calculated)	1.376 g/cm^3	
Absorption coefficient	0.554 mm^{-1}	
F(000)	688	
Crystal description	Needle	
Crystal color	Green	
Crystal size	0.15 x 0.05 x 0.05 mm^3	
Theta range for data collection	2.06 to 27.28	
Index ranges	-12<=h<=12, -11<=k<=14, -20<=l<=18	
Reflections collected	13588	
Independent reflections	6828 [R(int) = 0.1909]	
Completeness to theta = 27.28°	93.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6828 / 0 / 380	
Goodness-of-fit on F^2	0.999	
Final R indices [I>2sigma(I)]	R1 = 0.1294, wR2 = 0.3144	
R indices (all data)	R1 = 0.2400, wR2 = 0.3816	
Extinction coefficient	0.029(6)	
Largest diff. peak and hole	3.321 and -1.076 $e.Å^{-3}$	

Table 60.2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [TcO(L^{10}-COOEt)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Tc	6917(1)	8348(1)	7316(1)	41(1)
O(10)	6656(12)	9423(10)	6893(6)	50(2)
S(1)	9329(4)	9586(4)	7997(3)	58(1)
C(2)	9058(16)	10446(15)	8914(9)	48(4)
N(3)	7871(13)	9943(11)	9363(7)	43(3)
C(4)	6469(15)	8936(14)	9133(8)	37(3)
N(5)	5810(12)	8458(11)	8358(7)	41(3)
C(31)	5664(15)	8339(14)	9836(8)	38(3)
C(32)	5674(17)	9220(16)	10553(10)	51(4)
C(33)	4985(19)	8670(20)	11241(10)	60(5)
C(34)	4364(18)	7300(20)	11253(10)	59(4)
C(35)	4410(17)	6489(17)	10551(10)	54(4)
C(36)	5062(17)	6972(16)	9832(9)	48(4)
N(6)	10225(13)	11686(12)	9221(8)	47(3)
C(41)	10091(14)	12564(14)	9934(8)	40(3)
C(42)	8960(16)	12930(15)	9888(9)	47(3)
C(43)	8944(15)	13874(16)	10524(10)	48(4)
C(44)	9963(15)	14378(15)	11230(9)	48(4)
C(45)	11050(16)	13965(17)	11277(10)	56(4)
C(46)	11080(16)	13008(17)	10622(10)	51(4)
C(47)	11712(18)	12282(19)	8848(12)	63(4)
C(48)	9898(19)	15379(16)	11905(10)	52(4)
O(481)	8813(15)	15580(13)	11953(8)	74(3)
O(482)	11036(17)	15922(18)	12443(10)	101(5)
C(49)	11130(40)	17020(40)	13110(20)	190(20)
C(50)	12150(100)	17360(70)	13730(30)	400(60)
C(7)	4111(14)	7548(14)	8268(8)	41(3)
C(51)	3286(17)	7510(17)	7482(10)	52(4)
C(52)	2100(17)	7789(16)	7562(10)	52(4)
C(53)	1153(19)	7647(18)	6901(12)	67(5)
C(54)	1430(20)	7160(20)	6110(13)	79(6)
C(55)	2577(18)	6830(20)	5980(10)	62(4)
C(56)	3538(15)	7006(14)	6689(10)	44(3)
N(8)	4768(14)	6688(13)	6573(7)	47(3)

C(61)	5389(17)	6777(18)	5722(9)	52(4)
C(62)	6988(19)	6926(17)	5750(9)	54(4)
O(63)	7343(15)	6350(17)	5148(8)	91(5)
O(64)	7930(12)	7728(13)	6372(7)	59(3)
C(65)	4317(18)	5356(15)	6779(9)	48(3)
C(66)	5470(20)	5417(19)	7409(13)	69(5)
O(67)	5280(20)	4407(15)	7614(14)	132(8)
O(68)	6558(11)	6596(10)	7699(6)	51(2)

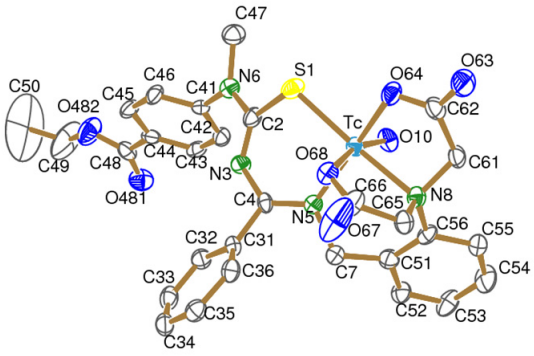

Figure 60 Ellipsoid plot (50% probability) of [TcO(L^{10}-COOEt)].