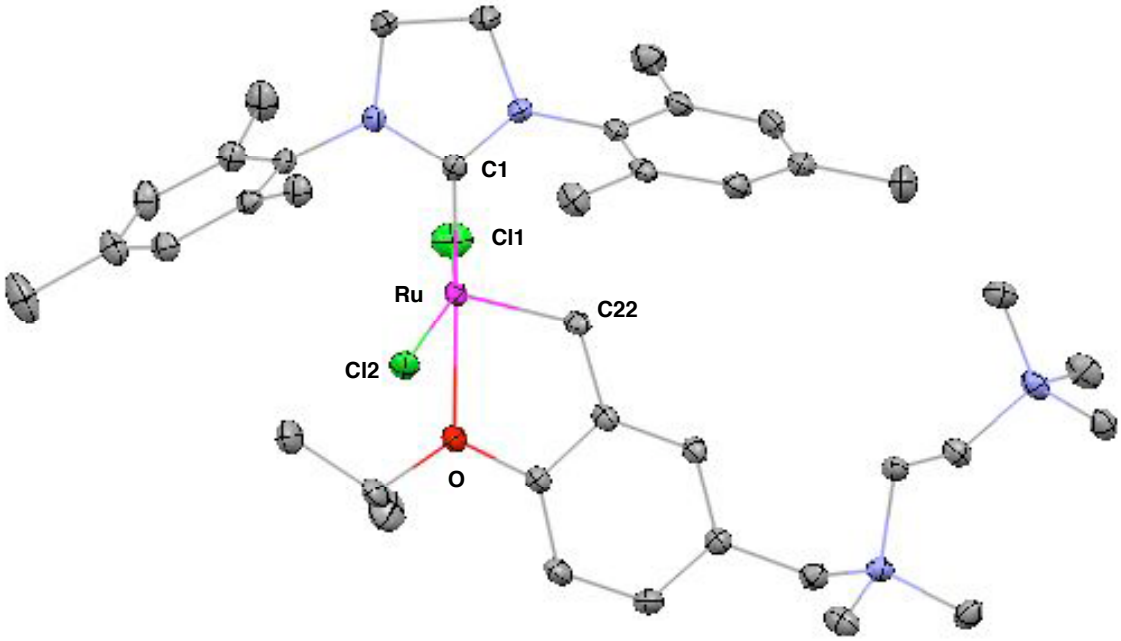
APPENDIX 2

Crystal Structure Data for Chapter 5



Selected bond lengths [Å] and angles [°] for JPJ02 (CCDC 623282)

Ru(1)-C(22)	1.8266(16)	C(22)-Ru(1)-C(1)	101.68(7)
Ru(1)-C(1)	1.9683(17)	C(22)-Ru(1)-O(1)	79.64(6)
Ru(1)-O(1)	2.2601(12)	C(1)-Ru(1)-O(1)	178.65(5)
Ru(1)-Cl(2)	2.3378(4)	C(22)-Ru(1)-Cl(2)	97.14(5)
Ru(1)-Cl(1)	2.3459(5)	C(1)-Ru(1)-Cl(2)	96.62(5)
		O(1)-Ru(1)-Cl(2)	82.94(3)
		C(22)-Ru(1)-Cl(1)	101.01(5)
		C(1)-Ru(1)-Cl(1)	91.65(5)
		O(1)-Ru(1)-Cl(1)	88.32(3)
		Cl(2)-Ru(1)-Cl(1)	158.086(18)

Crystal Data and Structure Refinement for JPJ02 (CCDC 623282)

Empirical Formula	$[C_{39}H_{62}N_4OCl_2Ru]^{+2}$ $2Cl^{-}$ • $2(CH_4O)$ • 0.14O
Formula Weight	908.06
Crystallization Solvent	Methanol/diethylether
Crystal Habit	Blade
Crystal Size	0.41 x 0.22 x 0.14 mm^3
Crystal Color	Green

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 25368 reflections used in lattice determination	2.24 to 37.53°
Unit cell dimensions	a = 27.8035(9) Å b = 12.0719(4) Å β = 104.1250(10)° c = 14.2362(4) Å
Volume	4633.8(3) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	P2/c
Density (calculated)	1.302 Mg/m^3
F(000)	1908.4
θ range for data collection	1.69° to 38.47°
Completeness to θ = 38.47°	87.9%
Index ranges	$-47 \le h \le 48$, $-17 \le k \le 19$, $-24 \le l \le 24$
Data collection scan type	ω scans at 5 ϕ settings
Reflections collected	83777
Independent reflections	22903 [R_{int}= 0.0907]
Absorption coefficient	0.608 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9197 and 0.7885

Structure Solution and Refinement

Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data/restraints/parameters	22903/0/501
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.219
Final R indices [I>2σ(I), 14094 reflections]	R1 = 0.0476, *w*R2 = 0.0816
R indices (all data)	R1 = 0.0914, *w*R2 = 0.0871
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.004
Average shift/error	0.000
Largest diff. peak and hole	1.995 and -1.110 e.Å^{-3}

Special Refinement Details

The Ru complex co-crystallizes with two molecules of methanol. The difference electron density Fourier contains a large peak on the 2-fold axis with no other nearby peaks. This peak was incorporated in the model as a site partially occupied by the oxygen of a water molecule. Least-squares refinement suggests 0.14 H_2O at this site forming a hydrogen bond to Cl4 at a distance of 3.1 Å.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.