APPENDIX B

X-RAY CRYSTAL DATA, REFINEMENT PARAMETERS AND MISCELLANEOUS STRUCTURES

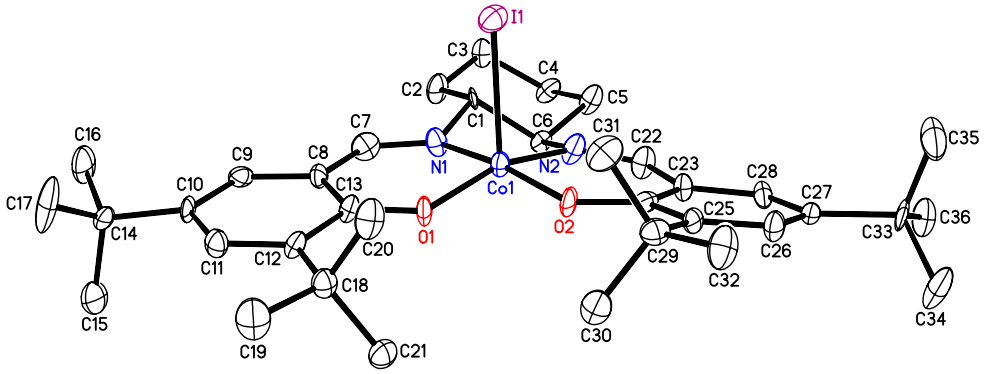

Figure B.1. ORTEP drawing of the (*R,R*)-(salen-**1**)CoI component of *rac*-(salen-**1**)CoI (non-hydrogen atoms) with thermal ellipsoids drawn at the 40% probability level. Selected bond lengths (Å) and bond angles (°): Co(1)-I(1), 2.714(1), Co(1)-O(1) 1.828(3), Co(1)-O(2) 1.858(3), Co(1)-N(1) 1.877(4), Co(1)-N(2) 1.866(4), O(1)-Co(1)-I(1) 100.6(1), O(2)-Co(1)-I(1) 95.8(1), N(1)-Co(1)-I(1) 92.8(1), N(2)-Co(1)-I(1) 98.2(1). The synthetic procedure for *rac*-(salen-**1**)CoI has been previously described in Chapter 2, however, *rac*-(salen-**1**)Co^{II} was used instead of (*R,R*)-(salen-**1**)Co^{II}. X-ray quality crystals of *rac*-(salen-**1**)CoI were obtained from slow evaporation of a methylene chloride/hexane (1:1) solution.

Table B.1. Crystal data and structure refinement for *rac*-(salen-**1**)CoI.

Identification code	*rac*-(salen-**1**)CoI	
Empirical formula	$C_{36}\ H_{52}\ Co\ I\ N_2\ O_2 \cdot CH_2Cl_2 \cdot 1/2(C_6H_{14})$	
Formula weight	858.64	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.969(4) Å	α= 97.299(9)°
	b = 12.522(5) Å	β= 105.834(9)°
	c = 16.694(7) Å	γ = 106.802(9)°
Volume	2058(1) $Å^3$	
Z	2	
Density (calculated)	1.385 mg/m^3	
Absorption coefficient	1.331 mm^{-1}	
F(000)	890	
Crystal size	0.40 x 0.15 x 0.05 mm^3	
Theta range for data collection	2.04 to 24.71°	
Index ranges	-12<=h<=11, -14<=k<=11, -19<=l<=19	
Reflections collected	10444	
Independent reflections	6823 [R(int) = 0.0491]	
Completeness to theta = 24.71°	97.2 %	
Absorption correction	SADABS	
Max. and min. transmission	0.9364 and 0.6181	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6823 / 159 / 537	
Goodness-of-fit on F^2	0.881	
Final R indices [I>2σ(I)]	R_1 = 0.0563, wR_2 = 0.1322	
R indices (all data)	R_1 = 0.0995, wR_2 = 0.1537	
Largest diff. peak and hole	0.690 and -0.847 $e \cdot Å^{-3}$	

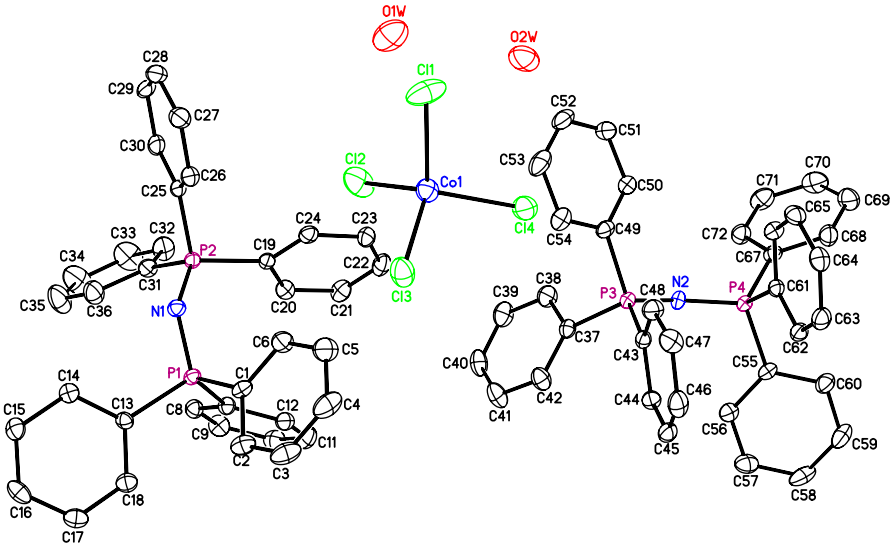

Figure B.2. ORTEP drawing of $[PPN]_2[CoCl_4]\cdot 2H_2O$ (non-hydrogen atoms) with thermal ellipsoids drawn at the 40% probability level. ([PPN] = $C_{36}H_{30}N_2P$). Synthetic procedure for $[PPN]_2[CoCl_4]\cdot 2H_2O$: In a drybox, a 1:1 mixture of [PPN]Cl and (*R,R*)-(salen-**1**)CoCl was added to a Schlenk. Following removal from the drybox, a minimal amount of methylene chloride was added under N_2 such that all of the solids dissolved. The solution was then carefully layered with diethyl ether under N_2. After two days, a bright blue solid was observed in the Schlenk, and the solution was opened to air, filtered, and washed with diethyl ether, yielding X-ray quality crystals of $[PPN]_2[CoCl_4]\cdot 2H_2O$.

Table B.2. Crystal data and structure refinement for $[PPN]_2[CoCl_4]\cdot 2H_2O$.

Identification code	$[PPN]_2[CoCl_4]\cdot 2H_2O$	
Empirical formula	$C_{72}\ H_{64}\ Cl_4\ Co\ N_2\ O_2\ P_4$	
Formula weight	1313.86	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$Pna2_1$	
Unit cell dimensions	a = 20.470(1) Å	α= 90°
	b = 23.619(1) Å	β= 90°
	c = 13.1693(7) Å	γ = 90°
Volume	6367.0(5) $Å^3$	
Z	4	
Density (calculated)	1.371 mg/m^3	
Absorption coefficient	0.586 mm^{-1}	
F(000)	2724	
Crystal size	0.40 x 0.30 x 0.10 mm^3	
Theta range for data collection	1.99 to 26.41°	
Index ranges	-25<=h<=25, -29<=k<=23, -16<=l<=16	
Reflections collected	92982	
Independent reflections	12933 [R(int) = 0.0366]	
Completeness to theta = 26.41°	99.9 %	
Absorption correction	Semiempirical by SADABS	
Max. and min. transmission	0.9437 and 0.7993	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12933 / 1 / 766	
Goodness-of-fit on F^2	1.017	
Final R indices [I>2σ(I)]	R_1 = 0.0458, wR_2 = 0.1293	
R indices (all data)	R_1 = 0.0529, wR_2 = 0.1359	
Absolute structure parameter	0.01(2)	
Largest diff. peak and hole	3.272 and -0.558 $e\cdot Å^{-3}$	

(*R,R*)-(salen-**1**)CoCl (**2.4**) (Chapter 2, Figure 2.8)

Table B.3. Crystal data and structure refinement for (*R,R*)-(salen-**1**)CoCl (**2.4**).

Identification code	(*R,R*)-(salen-**1**)CoCl	
Empirical formula	$C_{96}H_{128}Cl_2Co_2N_4O_4$	
Formula weight	1590.78	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 14.509(1) Å	α= 90°
	b = 10.1068(6) Å	β= 93.639(3)°
	c = 29.765(2) Å	γ = 90°
Volume	4355.9(5) $Å^3$	
Z	2	
Density (calculated)	1.213 mg/m^3	
Absorption coefficient	0.494 mm^{-1}	
F(000)	1704	
Crystal size	0.20 x 0.05 x 0.04 mm^3	
Theta range for data collection	1.52 to 20.87°	
Index ranges	-14<=h<=14, -10<=k<=10, -26<=l<=29	
Reflections collected	23614	
Independent reflections	8926 [R(int) = 0.0679]	
Completeness to theta = 20.87°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9805 and 0.9076	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8926 / 955 / 973	
Goodness-of-fit on F^2	1.076	
Final R indices [I>2σ(I)]	R_1 = 0.0589, wR_2 = 0.1325	
R indices (all data)	R_1 = 0.0808, wR_2 = 0.1408	
Absolute structure parameter	0.02(2)	
Largest diff. peak and hole	0.546 and -0.376 $e \cdot Å^{-3}$	