Supporting Information

Isolation of Di(η^2-nitrato)(η^4-cyclobutadiene)cobalt(II) Complexes from the Oxidation of Sterically Hindered CpCo-Stabilized Cyclobutadiene Complexes

Carsten Schaefer, Rolf Gleiter* and Frank Rominger

Organisch-Chemisches Institut der Universität Heidelberg,

Im Neuenheimer Feld 270, D-69120 Heidelberg, Germany

Table of Contents

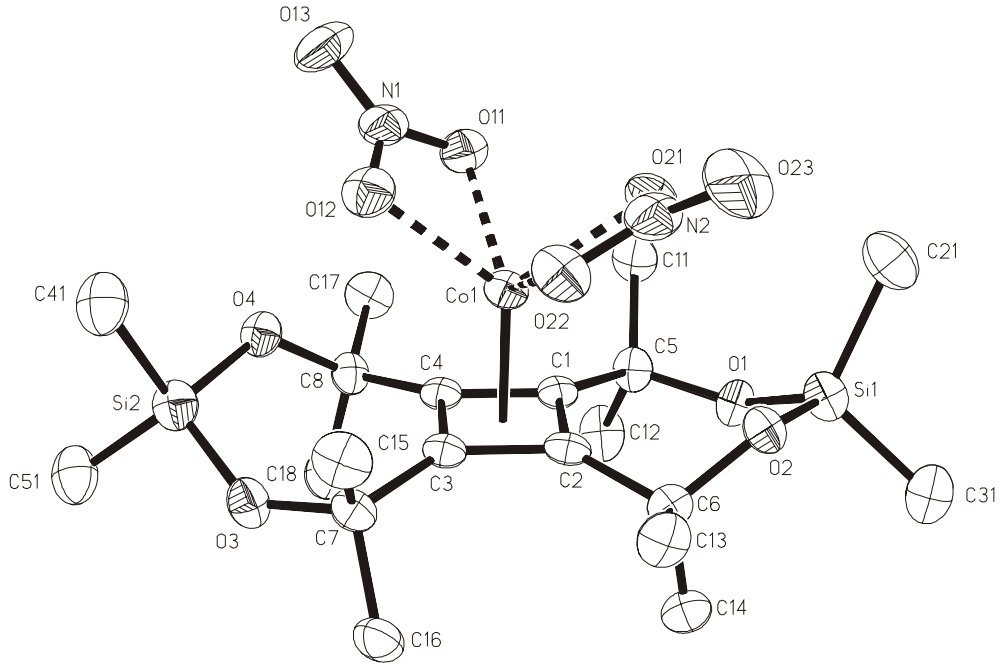

Table 1: Crystal data and structure refinement for **10a**.

Empirical formula	$C_{21}H_{38}Cl_2CoN_2O_{10}Si_2$	
Formula weight	664.54	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Z	4	
Unit cell dimensions	a = 11.3438(2) Å	α = 90 deg.
	b = 15.0766(4) Å	β = 94.560(1) deg.
	c = 17.6375(4) Å	γ = 90 deg.
Volume	3006.92(12) $Å^3$	
Density (calculated)	1.47 g/cm^3	
Absorption coefficient	0.88 mm^{-1}	
Crystal shape	lamina	
Crystal size	0.38 x 0.28 x 0.03 mm^3	
Theta range for data collection	1.8 to 22.0 deg.	
Index ranges	$-11 \le h \le 11$, $-15 \le k \le 15$, $-18 \le l \le 18$	
Reflections collected	19335	
Independent reflections	3668 (R(int) = 0.0768)	
Observed reflections	2632 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.97 and 0.73	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	3668 / 0 / 355	
Goodness-of-fit on F^2	1.02	
Final R indices ($I > 2\sigma(I)$)	R1 = 0.044, wR2 = 0.101	
Largest diff. peak and hole	0.67 and -0.54 $eÅ^{-3}$	

Table 2: Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **10a**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
Co1	0.5763(1)	0.6780(1)	0.3377(1)	0.0234(2)
Si1	0.9371(1)	0.6243(1)	0.4062(1)	0.0315(4)
O1	0.8584(3)	0.6365(2)	0.4790(2)	0.0279(8)
O2	0.8832(3)	0.6943(2)	0.3398(2)	0.0266(8)
Si2	0.3093(1)	0.8643(1)	0.3863(1)	0.0331(4)
O3	0.4311(3)	0.9138(2)	0.3688(2)	0.0324(9)
O4	0.3470(3)	0.7696(2)	0.4293(2)	0.0293(8)
N1	0.3810(4)	0.6010(3)	0.3208(2)	0.0344(11)
O11	0.4642(3)	0.5741(2)	0.3676(2)	0.0343(9)
O12	0.4073(3)	0.6709(2)	0.2835(2)	0.0367(9)
O13	0.2858(3)	0.5649(3)	0.3118(2)	0.0514(11)
N2	0.6825(4)	0.6153(3)	0.2327(3)	0.0397(12)
O21	0.6720(3)	0.5753(2)	0.2960(2)	0.0360(9)
O22	0.6256(3)	0.6882(2)	0.2257(2)	0.0369(9)
O23	0.7422(4)	0.5862(3)	0.1839(2)	0.0633(12)
C1	0.6714(4)	0.7059(3)	0.4387(2)	0.0211(11)
C2	0.7076(4)	0.7633(3)	0.3780(2)	0.0217(12)
C3	0.5914(4)	0.8081(3)	0.3736(2)	0.0210(11)
C4	0.5559(4)	0.7496(3)	0.4350(2)	0.0214(11)
C5	0.7378(4)	0.6419(3)	0.4940(3)	0.0267(12)
C6	0.8288(4)	0.7788(3)	0.3500(3)	0.0253(12)
C7	0.5372(4)	0.8906(3)	0.3358(3)	0.0272(12)
C8	0.4485(4)	0.7540(3)	0.4817(3)	0.0265(12)
C11	0.6836(4)	0.5496(3)	0.4911(3)	0.0355(14)
C12	0.7396(5)	0.6815(4)	0.5739(3)	0.0400(14)
C13	0.8298(4)	0.8251(3)	0.2732(3)	0.0330(13)
C14	0.8969(4)	0.8345(3)	0.4119(3)	0.0326(13)
C15	0.5134(5)	0.8785(3)	0.2495(3)	0.0358(13)
C16	0.6182(4)	0.9693(3)	0.3541(3)	0.0347(13)
C17	0.4200(5)	0.6705(3)	0.5236(3)	0.0357(13)
C18	0.4694(4)	0.8301(3)	0.5386(3)	0.0342(13)
C21	0.9338(5)	0.5120(3)	0.3634(3)	0.0464(15)
C31	1.0890(4)	0.6512(4)	0.4421(3)	0.0424(15)
C41	0.2122(5)	0.8351(4)	0.3018(3)	0.0439(15)
C51	0.2317(5)	0.9395(4)	0.4486(3)	0.0439(15)
C60	1.0602(7)	0.6552(4)	0.1876(4)	0.069(2)
Cl1	1.0700(2)	0.7411(1)	0.1234(1)	0.1071(8)
Cl2	1.0484(2)	0.5529(1)	0.1458(1)	0.0927(7)

Table 3: Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **10a**.

Atom	x	y	z	U_{eq}
H11A	0.6861	0.5253	0.4397	0.053
H11B	0.6014	0.5530	0.5041	0.053
H11C	0.7286	0.5110	0.5275	0.053
H12A	0.7887	0.6445	0.6095	0.060
H12B	0.6588	0.6838	0.5898	0.060
H12C	0.7724	0.7416	0.5736	0.060
H13A	0.9113	0.8295	0.2589	0.049
H13B	0.7963	0.8848	0.2767	0.049
H13C	0.7824	0.7909	0.2346	0.049
H14A	0.9020	0.8019	0.4601	0.049
H14B	0.8555	0.8907	0.4180	0.049
H14C	0.9768	0.8464	0.3969	0.049
H15A	0.4608	0.8277	0.2391	0.054
H15B	0.5883	0.8681	0.2268	0.054
H15C	0.4760	0.9322	0.2273	0.054
H16A	0.5812	1.0233	0.3323	0.052
H16B	0.6939	0.9595	0.3323	0.052
H16C	0.6316	0.9759	0.4094	0.052
H17A	0.3482	0.6797	0.5501	0.054
H17B	0.4860	0.6559	0.5608	0.054
H17C	0.4073	0.6216	0.4873	0.054
H18A	0.4830	0.8852	0.5111	0.051
H18B	0.5388	0.8168	0.5734	0.051
H18C	0.3999	0.8369	0.5677	0.051
H21A	0.9678	0.4692	0.4008	0.070
H21B	0.9800	0.5120	0.3187	0.070
H21C	0.8518	0.4955	0.3480	0.070
H31A	1.0916	0.7117	0.4626	0.064
H31B	1.1411	0.6471	0.4005	0.064
H31C	1.1155	0.6093	0.4824	0.064
H41A	0.1836	0.8894	0.2759	0.066
H41B	0.1447	0.8008	0.3172	0.066
H41C	0.2564	0.7996	0.2672	0.066
H51A	0.2897	0.9761	0.4786	0.066
H51B	0.1864	0.9044	0.4830	0.066
H51C	0.1778	0.9780	0.4173	0.066
H60A	1.1313	0.6561	0.2240	0.083
H60B	0.9905	0.6652	0.2168	0.083

Table 4: Anisotropic displacement parameters ($Å^2$) for **10a**. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Co1	0.0306(4)	0.0239(4)	0.0155(4)	-0.0018(3)	0.0003(3)	-0.0004(3)
Si1	0.0367(9)	0.0329(9)	0.0251(8)	0.0036(7)	0.0046(7)	0.0054(7)
O1	0.024(2)	0.037(2)	0.0227(19)	0.0055(15)	0.0024(15)	0.0017(16)
O2	0.033(2)	0.029(2)	0.0186(18)	-0.0006(14)	0.0049(15)	0.0046(16)
Si2	0.0352(9)	0.0381(9)	0.0256(8)	0.0013(7)	0.0006(7)	0.0040(7)
O3	0.033(2)	0.031(2)	0.033(2)	0.0050(16)	0.0062(17)	0.0064(16)
O4	0.028(2)	0.036(2)	0.0238(19)	-0.0001(16)	0.0001(16)	0.0017(16)
N1	0.035(3)	0.038(3)	0.031(3)	-0.016(2)	0.007(2)	-0.005(3)
O11	0.034(2)	0.035(2)	0.034(2)	-0.0023(17)	0.0016(18)	-0.0035(17)
O12	0.040(2)	0.041(2)	0.028(2)	-0.0008(19)	-0.0042(17)	-0.0008(18)
O13	0.032(2)	0.071(3)	0.052(3)	-0.019(2)	0.006(2)	-0.019(2)
N2	0.041(3)	0.047(3)	0.032(3)	-0.017(3)	0.007(2)	-0.009(3)
O21	0.045(2)	0.032(2)	0.032(2)	-0.0028(18)	0.0079(18)	0.0018(17)
O22	0.051(2)	0.042(2)	0.0182(19)	-0.0036(16)	0.0029(17)	0.004(2)
O23	0.075(3)	0.070(3)	0.048(3)	-0.027(2)	0.031(2)	0.000(2)
C1	0.026(3)	0.023(3)	0.014(3)	-0.006(2)	0.000(2)	-0.003(2)
C2	0.033(3)	0.019(3)	0.012(3)	-0.004(2)	0.000(2)	-0.004(2)
C3	0.029(3)	0.020(3)	0.014(2)	-0.002(2)	-0.001(2)	-0.002(2)
C4	0.028(3)	0.021(3)	0.015(3)	-0.003(2)	-0.003(2)	-0.003(2)
C5	0.030(3)	0.032(3)	0.018(3)	0.008(2)	-0.001(2)	0.004(2)
C6	0.028(3)	0.030(3)	0.018(3)	0.000(2)	0.002(2)	-0.002(2)
C7	0.033(3)	0.023(3)	0.026(3)	0.001(2)	0.003(2)	0.001(2)
C8	0.027(3)	0.031(3)	0.021(3)	-0.002(2)	0.002(2)	0.004(2)
C11	0.039(3)	0.030(3)	0.038(3)	0.017(3)	0.006(3)	0.002(3)
C12	0.044(3)	0.056(4)	0.020(3)	0.004(3)	0.001(3)	0.011(3)
C13	0.036(3)	0.039(3)	0.025(3)	0.010(3)	0.009(2)	-0.003(3)
C14	0.034(3)	0.032(3)	0.032(3)	0.003(2)	0.002(3)	-0.006(2)
C15	0.049(4)	0.037(3)	0.021(3)	0.009(2)	0.002(3)	0.000(3)
C16	0.040(3)	0.023(3)	0.042(3)	0.004(2)	0.007(3)	0.000(2)
C17	0.044(3)	0.039(3)	0.026(3)	0.007(3)	0.010(3)	-0.002(3)
C18	0.035(3)	0.043(3)	0.025(3)	-0.004(3)	0.008(2)	0.005(3)
C21	0.063(4)	0.038(4)	0.039(3)	0.004(3)	0.008(3)	0.011(3)
C31	0.037(3)	0.054(4)	0.037(3)	0.011(3)	0.005(3)	0.005(3)
C41	0.043(4)	0.056(4)	0.032(3)	0.003(3)	-0.002(3)	0.008(3)
C51	0.042(4)	0.054(4)	0.035(3)	0.000(3)	-0.003(3)	0.011(3)
C60	0.108(6)	0.048(4)	0.052(4)	-0.001(3)	0.010(4)	0.006(4)
Cl1	0.171(2)	0.0864(15)	0.0601(13)	0.0196(11)	-0.0145(14)	-0.0173(14)
Cl2	0.0913(15)	0.0703(13)	0.1163(17)	-0.0390(12)	0.0071(12)	0.0006(11)

Table 5: Bond lengths (Å) and angles (deg) for **10a**.

Co1-C1	2.051(4)
Co1-C2	2.051(4)
Co1-C4	2.056(4)
Co1-O21	2.060(3)
Co1-C3	2.063(4)
Co1-O12	2.076(3)
Co1-O22	2.102(3)
Co1-O11	2.110(3)
Si1-O1	1.633(3)
Si1-O2	1.657(3)
Si1-C31	1.834(5)
Si1-C21	1.852(5)
O1-C5	1.416(5)
O2-C6	1.433(5)
Si2-O3	1.622(3)
Si2-O4	1.656(3)
Si2-C41	1.835(5)
Si2-C51	1.851(5)
O3-C7	1.421(5)
O4-C8	1.438(5)
N1-O13	1.209(5)
N1-O11	1.270(5)
N1-O12	1.289(5)
N2-O23	1.218(5)
N2-O22	1.276(5)
N2-O21	1.282(5)
C1-C2	1.460(6)
C1-C4	1.464(6)
C1-C5	1.526(6)
C2-C3	1.478(6)
C2-C6	1.515(7)
C3-C4	1.477(6)
C3-C7	1.519(6)
C4-C8	1.525(6)
C5-C11	1.520(6)
C5-C12	1.529(7)
C6-C13	1.526(6)
C6-C14	1.536(6)
C7-C16	1.519(6)
C7-C15	1.537(6)
C8-C17	1.509(6)
C8-C18	1.530(6)
C60-Cl2	1.710(6)
C60-Cl1	1.729(6)
C1-Co1-C2	41.72(17)
C1-Co1-C4	41.79(17)
C2-Co1-C4	60.59(18)
C1-Co1-O21	102.00(16)
C2-Co1-O21	101.87(16)
C4-Co1-O21	142.22(16)
C1-Co1-C3	60.93(17)
C2-Co1-C3	42.10(17)
C4-Co1-C3	42.01(17)
O21-Co1-C3	142.23(17)
C1-Co1-O12	144.44(16)
C2-Co1-O12	144.13(16)
C4-Co1-O12	104.50(16)
O21-Co1-O12	106.88(13)

C3-Co1-O12	103.97(16)
C1-Co1-O22	129.83(16)
C2-Co1-O22	92.27(16)
C4-Co1-O22	143.37(16)
O21-Co1-O22	62.08(14)
C3-Co1-O22	101.38(16)
O12-Co1-O22	82.85(14)
C1-Co1-O11	102.79(16)
C2-Co1-O11	144.51(16)
C4-Co1-O11	94.11(15)
O21-Co1-O11	82.91(13)
C3-Co1-O11	131.74(16)
O12-Co1-O11	61.57(14)
O22-Co1-O11	119.95(13)
O1-Si1-O2	106.87(17)
O1-Si1-C31	105.1(2)
O2-Si1-C31	112.7(2)
O1-Si1-C21	115.4(2)
O2-Si1-C21	107.4(2)
C31-Si1-C21	109.4(3)
C5-O1-Si1	138.6(3)
C6-O2-Si1	128.0(3)
O3-Si2-O4	106.93(17)
O3-Si2-C41	114.9(2)
O4-Si2-C41	106.3(2)
O3-Si2-C51	106.5(2)
O4-Si2-C51	112.0(2)
C41-Si2-C51	110.2(2)
C7-O3-Si2	136.6(3)
C8-O4-Si2	126.8(3)
O13-N1-O11	123.5(5)
O13-N1-O12	122.8(5)
O11-N1-O12	113.7(4)
N1-O11-Co1	91.8(3)
N1-O12-Co1	92.9(3)
O23-N2-O22	123.1(5)
O23-N2-O21	122.8(5)
O22-N2-O21	114.1(4)
N2-O21-Co1	92.6(3)
N2-O22-Co1	90.9(3)
C2-C1-C4	90.2(4)
C2-C1-C5	133.6(4)
C4-C1-C5	135.0(4)
C2-C1-Co1	69.2(2)
C4-C1-Co1	69.3(2)
C5-C1-Co1	128.6(3)
C1-C2-C3	90.4(4)
C1-C2-C6	130.2(4)
C3-C2-C6	137.8(4)
C1-C2-Co1	69.1(2)
C3-C2-Co1	69.4(2)
C6-C2-Co1	129.6(3)
C4-C3-C2	89.0(3)
C4-C3-C7	133.3(4)
C2-C3-C7	136.5(4)
C4-C3-Co1	68.7(2)
C2-C3-Co1	68.5(2)
C7-C3-Co1	128.6(3)
C1-C4-C3	90.3(4)
C1-C4-C8	138.3(4)
C3-C4-C8	130.3(4)

C1-C4-Co1	68.9(2)
C3-C4-Co1	69.3(2)
C8-C4-Co1	128.4(3)
O1-C5-C11	109.5(4)
O1-C5-C1	110.6(4)
C11-C5-C1	112.3(4)
O1-C5-C12	104.6(4)
C11-C5-C12	111.4(4)
C1-C5-C12	108.2(4)
O2-C6-C2	108.4(4)
O2-C6-C13	105.2(4)
C2-C6-C13	115.5(4)
O2-C6-C14	112.2(4)
C2-C6-C14	105.8(4)
C13-C6-C14	110.0(4)
O3-C7-C3	110.4(4)
O3-C7-C16	103.8(4)
C3-C7-C16	109.3(4)
O3-C7-C15	110.3(4)
C3-C7-C15	111.8(4)
C16-C7-C15	111.0(4)
O4-C8-C17	105.1(4)
O4-C8-C4	107.1(4)
C17-C8-C4	116.1(4)
O4-C8-C18	112.0(4)
C17-C8-C18	109.4(4)
C4-C8-C18	107.3(4)
Cl2-C60-Cl1	113.6(4)