CRYSTAL STRUCTURE ANALYSIS REPORT and TABLES for

(3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato)cobalt(III) (38)

Wake Forest X-Ray Facility Reference Code: **MWE7-0999 / BH200**

For: **Prof. M. Welker**

Single crystals of $[O_2N_2C_4H_7]_2Co(NC_5H_5)(C_6OH_9)$ are, at -80±1°C, monoclinic, space group Pn (an alternate setting of Pc - C_s^2 (No.7)) with a = 8.3411(8)Å, b = 11.774(1)Å, c = 10.913(1)Å, ß= 94.202(2)°, V = 1068.88(18)Å^3, and Z = 2 {d_{calcd} = 1.446gcm^{-3}; μ_a(MoK $\bar{\alpha}$) = 0.843 mm^{-1}}. A full hemisphere of diffracted intensities (omega scan width of 0.25°) was measured using graphite-monochromated MoK$\bar{\alpha}$ radiation on a Bruker SMART CCD area detector. Xrays were provided by a normal focus sealed X-ray tube operated at 50kV and 40mA. Lattice constants were determined with the Bruker SMART software package using peak centers for 1880 reflections. A total of 5654 integrated reflection intensities having 2θ(MoK$\bar{\alpha}$)< 50.1° were collected using the Bruker program SAINT. A total of 2649 of these were independent and gave R_{int} = 0.052. The Bruker SHELXTL-PC software package was used to solve the structure using "Direct Methods" techniques. All stages of weighted full-matrix least-squares refinement were conducted using F_o^2 data. The resulting structural parameters have been refined to convergence {R_1 (unweighted, based on F) = 0.0439 for 1874 independent reflections having 2θ(MoK$\bar{\alpha}$ < 50.1° and $F^2>2\sigma(F^2)$ } {R_1 (unweighted, based on F) = 0.0715 and wR_2 (weighted, based on F^2) = 0.0948 for all 2649 reflections} using counter-weighted full-matrix least-squares techniques and a structural model which incorporated anisotropic thermal parameters for all nonhydrogen atoms. Hydrogen atoms H_{2o}, H_{4o}, H_{14a} and H_{14b} were located from a difference Fourier map and refined as independent isotropic atoms (except H_{4o} which was included in the structural model at its difference Fourier positions).

The five methyl groups were refined as rigid rotors with sp^3-hybridized geometry and a C-H bond length of 0.98Å. The initial orientation for each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation for each of these methyl groups was determined by one torsion angle parameter per methyl group. The remaining hydrogen atoms not on solvent molecules were included in the structure factor calculations as idealized atoms (assuming sp^3- or sp^2-hybridization of the carbon atoms and C-H bond lengths of 0.95 - 0.99Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{2o}, H_{4o}, H_{14a} and H_{14b} refined to final U_{iso} values of 0.10(3), 0.08(4), 0.02(1), and 0.05(2)Å^2, respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (aromatic or methylene) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

The absolute structure (Flack) parameter was varied during the final cycle of refinement; its final value was 0.35(3). This value suggests the presence of an inversion twin since expected values are 0(within 3 esd's) for correct and +1 for inverted absolute structures.

All calculations were performed using the SHELXTL-PC (Version 5.1) interactive software package (G. Sheldrick, Bruker-AXS, Madison, WI).

Table 1. Crystal data and structure refinement for (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)

Empirical formula	$C_{19}H_{28}CoN_5O_5$
Formula weight	465.39
Temperature	193(2)K
Wavelength	0.71073Å
Crystal system, space group description of Pc - C_s^2 (No.7))	Monoclinic, Pn (an alternate
Unit cell dimensions	a = 8.3411(8)Å b = 11.774(1)Å, ß = 94.202(2)° c = 10.913(1)Å
Volume	1068.88(18)Å^3
Z, Calculated density	2, 1.446g/cm^{-1}
Absorption coefficient	0.843 mm^{-1}
F(000)	488
Crystal size	0.13 x 0.14 x 0.24 mm
Theta range for data collection	1.73 to 25.05 deg.
Limiting indices	$-9\le h\le 9$, $-14\le k\le 12$, $-12\le l\le 8$
Reflections collected / unique	5654 / 2649 [R_{int} = 0.0523]
Completeness to theta = 25.05	99.8 %
Absorption correction	Empirical
Max. and min. transmission	0.5287 and 0.4370
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2649 / 2 / 290
Goodness-of-fit on F^2	0.933
Final R indices	
[1874 F_o>4sigma(F_o) data]	R_1 = 0.0439, wR_2 = 0.0867
[all 2649 data]	R_1 = 0.0715, wR_2 = 0.0948
Absolute structure (Flack) parameter	0.35(3)
Largest diff. peak and hole	0.440 and -0.274 e$^-$/Å^3

Table 2. Atomic Coordinates for Atoms in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)[a]

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, Å^2 x 10^3 [c]
Co_1	1959(2)	2385(1)	-1593(1)	32(1)
O_1	2851(6)	4629(4)	-2226(5)	49(2)
O_2	3692(6)	326(4)	-1930(4)	45(2)
O_3	1084(7)	125(4)	-985(4)	45(1)
O_4	287(7)	4423(4)	-1191(5)	47(2)
O_5	-1969(7)	2868(5)	-5444(5)	87(2)
N_1	3238(7)	3507(5)	-2235(5)	32(2)
N_2	3621(7)	1452(5)	-2112(5)	33(2)
N_3	638(8)	1227(5)	-990(5)	33(2)
N_4	278(8)	3298(5)	-1106(5)	37(2)
N_5	3136(8)	2556(4)	113(6)	28(2)
C_1	4501(10)	3183(7)	-2766(7)	42(2)
C_2	4765(10)	1964(8)	-2673(6)	39(2)
C_3	5567(9)	4004(7)	-3367(7)	61(3)
C_4	6142(9)	1355(7)	-3158(8)	58(2)
C_5	-671(10)	1565(6)	-554(7)	36(2)
C_6	-897(9)	2789(7)	-620(6)	38(2)
C_7	-1840(9)	769(7)	-34(7)	69(3)
C_8	-2299(9)	3416(7)	-174(7)	63(3)
C_9	3365(8)	3590(6)	625(6)	38(2)
C_{10}	4127(10)	3741(6)	1782(8)	57(2)
C_{11}	4667(9)	2824(7)	2447(7)	59(2)
C_{12}	4433(9)	1763(6)	1963(7)	52(2)

Table 2. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, $Å^2 \times 10^3$ [c]
C_{13}	3660(9)	1641(6)	810(7)	45(2)
C_{14}	524(11)	1175(7)	-3756(8)	55(2)
C_{15}	822(11)	2203(7)	-3268(8)	43(2)
C_{16}	154(10)	3212(7)	-3839(7)	57(3)
C_{17}	-539(12)	3045(8)	-5177(8)	59(2)
C_{18}	627(10)	3053(7)	-6140(7)	74(2)
C_{19}	3(9)	2535(6)	-7340(6)	72(2)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] This is one-third of the trace of the orthogonalized U_{ij} tensor.

Table 3. Bond Lengths in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato)cobalt(III)[a]

Type[b]	Length, Å	Type[b]	Length, Å
Co_1-N_1	1.867(6)	Co_1-N_3	1.900(6)
Co_1-N_2	1.887(6)	Co_1-N_4	1.875(6)
Co_1-N_5	2.050(6)	Co_1-C_{15}	2.008(9)
O_1-N_1	1.359(8)	O_3-N_3	1.350(8)
O_2-N_2	1.342(8)	O_4-N_4	1.328(7)
O_5-C_{17}	1.225(10)		
N_1-C_1	1.296(9)	N_3-C_5	1.286(9)
N_2-C_2	1.316(9)	N_4-C_6	1.295(9)
N_5-C_9	1.347(8)	N_5-C_{13}	1.371(8)
C_1-C_2	1.454(14)	C_5-C_6	1.454(10)
C_1-C_3	1.496(10)	C_5-C_7	1.494(10)
C_2-C_4	1.485(11)	C_6-C_8	1.494(10)
C_9-C_{10}	1.382(9)	C_{15}-C_{16}	1.435(11)
C_{10}-C_{11}	1.359(9)	C_{16}-C_{17}	1.542(10)
C_{11}-C_{12}	1.365(11)	C_{17}-C_{18}	1.484(11)
C_{12}-C_{13}	1.379(9)		
C_{14}-C_{15}	1.339(11)	C_{18}-C_{19}	1.502(9)

Table 3. (continued)

Type[b]	Length, Å	Type[b]	Length, Å
O_2-H_{2o}	1.20(8)	O_4-H_{4o}[c]	0.85(-)
C_{14}-H_{14a}	1.20(5)	C_{14}-H_{14b}	0.93(6)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] Hydrogen atom H_{4o} was included in the structural model at fixed positions obtained from a difference Fourier map. Values listed for distances and angles involving this hydrogen therefore do not include estimated standard deviations.

Table 4. Bond Angles in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato)cobalt(III)[a]

Type[b,c]	Angle, (deg)	Type[b,c]	Angle, (deg)
N_1-Co_1-N_4	99.2(3)	N_1-Co_1-N_5	91.3(2)
N_1-Co_1-N_2	81.2(3)	N_4-Co_1-N_5	90.3(2)
N_4-Co_1-N_3	81.3(3)	N_2-Co_1-N_5	91.0(2)
N_2-Co_1-N_3	98.3(2)	N_3-Co_1-N_5	90.4(2)
N_1-Co_1-C_{15}	88.7(3)	C_{15}-Co_1-N_5	179.4(4)
N_4-Co_1-C_{15}	90.3(3)	N_4-Co_1-N_2	178.7(3)
N_2-Co_1-C_{15}	88.5(3)	N_1-Co_1-N_3	178.2(3)
N_3-Co_1-C_{15}	89.5(3)		
C_1-N_1-O_1	119.4(6)	C_5-N_3-O_3	122.5(6)
C_1-N_1-Co_1	117.7(6)	C_5-N_3-Co_1	115.9(5)
O_1-N_1-Co_1	122.8(5)	O_3-N_3-Co_1	121.6(5)
C_2-N_2-O_2	119.7(7)	C_6-N_4-O_4	119.9(7)
C_2-N_2-Co_1	116.5(6)	C_6-N_4-Co_1	117.1(5)
O_2-N_2-Co_1	123.7(5)	O_4-N_4-Co_1	123.0(6)
C_9-N_5-Co_1	120.7(4)	C_9-N_5-C_{13}	116.7(6)
C_{13}-N_5-Co_1	122.6(4)		
N_1-C_1-C_2	112.5(7)	N_3-C_5-C_6	113.5(7)
N_1-C_1-C_3	122.3(8)	N_3-C_5-C_7	122.8(7)
C_2-C_1-C_3	125.2(7)	C_6-C_5-C_7	123.7(7)
N_2-C_2-C_1	111.9(7)	N_4-C_6-C_5	112.3(7)
N_2-C_2-C_4	123.4(8)	N_4-C_6-C_8	122.6(8)
C_1-C_2-C_4	124.7(7)	C_5-C_6-C_8	125.1(7)

Table 4. (continued)

Type[b,c]	Angle, (deg)	Type[b,c]	Angle, (deg)
N_5-C_9-C_{10}	122.5(7)	C_{14}-C_{15}-C_{16}	121.4(8)
C_{11}-C_{10}-C_9	119.8(7)	C_{14}-C_{15}-Co_1	121.3(6)
C_{10}-C_{11}-C_{12}	119.2(7)	C_{16}-C_{15}-Co_1	116.8(6)
C_{11}-C_{12}-C_{13}	119.5(6)	O_5-C_{17}-C_{18}	120.6(8)
N_5-C_{13}-C_{12}	122.3(6)	O_5-C_{17}-C_{16}	122.6(8)
		C_{18}-C_{17}-C_{16}	116.8(8)
C_{15}-C_{16}-C_{17}	114.4(6)		
C_{17}-C_{18}-C_{19}	114.4(7)		
N_2-O_2-H_{2o}	101(3)	C_{15}-C_{14}-H_{14a}	116(2)
N_4-O_4-H_{4o}[c]	106(3)	C_{15}-C_{14}-H_{14b}	113(4)
		H_{14a}-C_{14}-H_{14b}	130(4)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] Hydrogen atom H_{4o} was included in the structural model at fixed positions obtained from a difference Fourier map. Values listed for distances and angles involving this hydrogen therefore do not include estimated standard deviations.

Table 5. Anisotropic Thermal Parameters for Nonhydrogen Atoms in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine) bis(dimethylglyoximato)cobalt(III)[a,b]

Atom Type[c]	Anisotropic Thermal Parameters ($Å^2$ x 10^3)					
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Co_1	30(1)	39(1)	27(1)	-1(1)	3(1)	-2(1)
O_1	63(4)	30(3)	56(4)	4(2)	10(3)	-2(3)
O_2	49(4)	38(3)	48(3)	-7(2)	11(3)	0(2)
O_3	60(4)	32(3)	44(3)	0(2)	6(3)	-8(2)
O_4	49(4)	46(3)	46(4)	4(2)	6(3)	14(3)
O_5	43(3)	160(5)	58(4)	8(3)	-5(3)	3(4)
N_1	36(4)	36(4)	23(3)	-1(2)	6(3)	-3(3)
N_2	30(4)	38(3)	31(4)	-7(3)	-2(3)	-2(3)
N_3	38(4)	32(4)	29(4)	-1(3)	3(3)	-6(3)
N_4	41(5)	33(3)	36(4)	6(2)	-3(3)	10(3)
N_5	24(4)	39(3)	20(3)	-4(2)	-2(3)	-3(2)
C_1	37(6)	51(5)	38(5)	2(4)	5(4)	-5(4)
C_2	32(5)	57(5)	29(5)	-9(4)	5(4)	-2(5)
C_3	58(6)	71(6)	57(6)	13(4)	27(5)	-16(4)
C_4	35(5)	90(6)	50(5)	-14(4)	9(4)	13(4)
C_5	29(5)	46(5)	33(5)	0(3)	-3(4)	-3(3)
C_6	21(4)	73(6)	21(4)	7(4)	-1(3)	3(5)
C_7	49(6)	89(7)	71(6)	4(5)	16(4)	-28(5)
C_8	46(6)	93(7)	50(5)	8(4)	12(4)	11(5)
C_9	39(5)	40(4)	37(5)	-11(3)	3(4)	-11(3)
C_{10}	54(5)	52(5)	63(6)	-12(4)	-2(4)	-8(4)
C_{11}	40(5)	95(7)	41(5)	-25(5)	0(4)	6(4)
C_{12}	61(6)	56(5)	37(5)	8(4)	-10(4)	26(4)
C_{13}	53(6)	48(4)	33(5)	2(3)	0(4)	16(4)

Table 5. (continued)

Atom Type[c]	Anisotropic Thermal Parameters ($Å^2$ x 10^3)					
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C_{14}	64(7)	60(5)	39(5)	0(4)	-7(4)	9(4)
C_{15}	50(6)	35(4)	44(6)	-2(4)	-3(4)	2(4)
C_{16}	67(7)	70(6)	33(5)	-9(4)	-3(4)	-1(5)
C_{17}	65(7)	70(5)	40(6)	2(4)	2(5)	21(5)
C_{18}	70(6)	105(6)	49(5)	13(4)	16(4)	-1(4)
C_{19}	81(6)	92(6)	44(4)	-18(4)	14(4)	-10(5)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] The form of the anisotropic thermal parameter is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

[c] Atoms are labeled in agreement with Figure 1.

Table 6. Atomic Coordinates for Hydrogen Atoms in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethyl glyoximato)cobalt(III)[a]

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z
H_{2o}[c]	2390(100)	140(60)	-1580(70)
H_{4o}	1199	4599	-1443
H_{3a}	5936	4590	-2774
H_{3b}	4965	4361	-4070
H_{3c}	6497	3599	-3651
H_{4a}	6067	543	-2971
H_{4b}	7151	1659	-2774
H_{4c}	6118	1460	-4050
H_{7a}	-1568	-14	-244
H_{7b}	-2929	945	-380
H_{7c}	-1789	853	861
H_{8a}	-2637	4012	-765
H_{8b}	-1990	3760	626
H_{8c}	-3191	2886	-91
H_9	2990	4239	175
H_{10}	4272	4484	2111
H_{11}	5202	2921	3239
H_{12}	4800	1113	2417
H_{13}	3484	899	487
H_{14a}[c]	-60(60)	1160(40)	-4790(50)
H_{14b}[c]	880(70)	580(50)	-3240(60)
H_{16a}	1001	3802	-3827
H_{16b}	-711	3498	-3346
H_{18a}	1604	2635	-5830

Table 6. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z
H_{18b}	945	3848	-6289
H_{19a}	745	2707	-7971
H_{19b}	-1058	2852	-7587
H_{19c}	-83	1711	-7245

[a] Hydrogen atoms H_{2o}, H_{4o}, H_{14a} and H_{14b} were located a difference Fourier map and were refined as independent isotropic atoms (with the exception of H_{4o} whose coordinates were fixed at their difference Fourier values). The five methyl groups were refined as rigid rotors with sp^3-hybridized geometry and a C-H bond length of 0.98Å. The initial orientation for each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation for each of these methyl groups was determined by one torsion angle parameter per methyl group. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp^3- or sp^2-hybridization of the carbon atoms and C-H bond lengths of 0.95 - 0.99Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{2o}, H_{4o}, H_{14a} and H_{14b} refined to final values of 0.10(3), 0.08(4), 0.02(1), and 0.05(2)Å^2, respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (aromatic or methylene) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

[b] Hydrogen atoms which are covalently bonded to carbon are labeled with the same numerical subscript(s) as their carbon atoms with an additional literal subscript (a, b or c) where necessary to distinguish between hydrogens bonded to the same carbon atom.

[c] The numbers in parentheses are the estimated standard deviations in the last significant digit.

Table 7. Torsion angles [deg] for (3-Oxo-5-hexen-5-yl)(pyridine) bis(dimethylglyoximato)cobalt(III)[a]

Type[b]	Angle, (deg)	Type[b]	Angle, (deg)
N_4-Co_1-N_1-C_1	-174.7(5)	N_2-Co_1-N_3-O_3	2.8(6)
N_2-Co_1-N_1-C_1	4.0(6)	C_{15}-Co_1-N_3-O_3	91.2(5)
N_3-Co_1-N_1-C_1	-69(11)	N_5-Co_1-N_3-O_3	-88.2(5)
C_{15}-Co_1-N_1-C_1	-84.6(6)	N_1-Co_1-N_4-C_6	178.8(5)
N_5-Co_1-N_1-C_1	94.8(6)	N_2-Co_1-N_4-C_6	72(15)
N_4-Co_1-N_1-O_1	1.6(6)	N_3-Co_1-N_4-C_6	0.6(5)
N_2-Co_1-N_1-O_1	-179.6(5)	C_{15}-Co_1-N_4-C_6	90.0(6)
N_3-Co_1-N_1-O_1	108(11)	N_5-Co_1-N_4-C_6	-89.8(5)
C_{15}-Co_1-N_1-O_1	91.7(5)	N_1-Co_1-N_4-O_4	-4.2(6)
N_5-Co_1-N_1-O_1	-88.9(5)	N_2-Co_1-N_4-O_4	-111(15)
N_1-Co_1-N_2-C_2	-2.4(6)	N_3-Co_1-N_4-O_4	177.5(5)
N_4-Co_1-N_2-C_2	104(15)	C_{15}-Co_1-N_4-O_4	-93.0(5)
N_3-Co_1-N_2-C_2	175.9(5)	N_5-Co_1-N_4-O_4	87.1(5)
C_{15}-Co_1-N_2-C_2	86.6(6)	N_1-Co_1-N_5-C_9	49.1(6)
N_5-Co_1-N_2-C_2	-93.5(5)	N_4-Co_1-N_5-C_9	-50.1(6)
N_1-Co_1-N_2-O_2	177.1(5)	N_2-Co_1-N_5-C_9	130.3(6)
N_4-Co_1-N_2-O_2	-76(15)	N_3-Co_1-N_5-C_9	-131.4(6)
N_3-Co_1-N_2-O_2	-4.7(6)	C_{15}-Co_1-N_5-C_9	142(39)
C_{15}-Co_1-N_2-O_2	-94.0(6)	N_1-Co_1-N_5-C_{13}	-133.9(6)
N_5-Co_1-N_2-O_2	85.9(5)	N_4-Co_1-N_5-C_{13}	126.9(7)
N_1-Co_1-N_3-C_5	-107(11)	N_2-Co_1-N_5-C_{13}	-52.7(7)
N_4-Co_1-N_3-C_5	-0.6(6)	N_3-Co_1-N_5-C_{13}	45.6(7)
N_2-Co_1-N_3-C_5	-179.4(5)	C_{15}-Co_1-N_5-C_{13}	-42(39)
C_{15}-Co_1-N_3-C_5	-91.0(6)	O_1-N_1-C_1-C_2	178.8(5)
N_5-Co_1-N_3-C_5	89.5(5)	Co_1-N_1-C_1-C_2	-4.7(8)
N_1-Co_1-N_3-O_3	75(11)	O_1-N_1-C_1-C_3	0.5(11)

Table 7. (continued)

Type[b]	Angle, (deg)	Type[b]	Angle, (deg)
N_4-Co_1-N_3-O_3	-178.4(5)	Co_1-N_1-C_1-C_3	176.9(5)
O_2-N_2-C_2-C_1	-178.9(5)	N_5-C_9-C_{10}-C_{11}	-0.3(12)
Co_1-N_2-C_2-C_1	0.5(7)	C_9-C_{10}-C_{11}-C_{12}	-0.7(12)
O_2-N_2-C_2-C_4	1.7(11)	C_{10}-C_{11}-C_{12}-C_{13}	0.2(11)
Co_1-N_2-C_2-C_4	-178.9(6)	C_9-N_5-C_{13}-C_{12}	-2.1(12)
N_1-C_1-C_2-N_2	2.6(8)	Co_1-N_5-C_{13}-C_{12}	-179.2(5)
C_3-C_1-C_2-N_2	-179.1(7)	C_{11}-C_{12}-C_{13}-N_5	1.2(12)
N_1-C_1-C_2-C_4	-178.0(7)	N_1-Co_1-C_{15}-C_{14}	134.3(9)
C_3-C_1-C_2-C_4	0.3(10)	N_4-Co_1-C_{15}-C_{14}	-126.5(9)
O_3-N_3-C_5-C_6	178.4(6)	N_2-Co_1-C_{15}-C_{14}	53.1(9)
Co_1-N_3-C_5-C_6	0.6(8)	N_3-Co_1-C_{15}-C_{14}	-45.2(9)
O_3-N_3-C_5-C_7	-1.8(11)	N_5-Co_1-C_{15}-C_{14}	42(40)
Co_1-N_3-C_5-C_7	-179.5(5)	N_1-Co_1-C_{15}-C_{16}	-54.0(7)
O_4-N_4-C_6-C_5	-177.5(5)	N_4-Co_1-C_{15}-C_{16}	45.2(7)
Co_1-N_4-C_6-C_5	-0.4(8)	N_2-Co_1-C_{15}-C_{16}	-135.2(7)
O_4-N_4-C_6-C_8	2.0(10)	N_3-Co_1-C_{15}-C_{16}	126.5(7)
Co_1-N_4-C_6-C_8	179.0(5)	N_5-Co_1-C_{15}-C_{16}	-146(38)
N_3-C_5-C_6-N_4	-0.1(9)	C_{14}-C_{15}-C_{16}-C_{17}	-14.7(14)
C_7-C_5-C_6-N_4	180.0(7)	Co_1-C_{15}-C_{16}-C_{17}	173.6(6)
N_3-C_5-C_6-C_8	-179.5(7)	C_{15}-C_{16}-C_{17}-O_5	96.1(11)
C_7-C_5-C_6-C_8	0.6(11)	C_{15}-C_{16}-C_{17}-C_{18}	-81.1(11)
C_{13}-N_5-C_9-C_{10}	1.7(12)	O_5-C_{17}-C_{18}-C_{19}	-15.4(13)
Co_1-N_5-C_9-C_{10}	178.8(5)	C_{16}-C_{17}-C_{18}-C_{19}	161.8(7)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

Table 8. Hydrogen-Bonding Interactions in Crystalline (3-Oxo-5-hexen-5-yl)(pyridine)bis(dimethylglyoximato)cobalt(III)[a]

Donor[b] Atom D	Acceptor Atom A	Distance Å D···A[c]	Distance Å H···A[c]	Angle, deg. D-H···A[c]	Angle, deg. H-D···A[c]	Angle, deg. H···A···X[c]	Asymmetric[d] Unit of A
O_2-H_{2o}	O_3	2.486(7)	1.31(9)	165(6)	8(3)	103(3) N_3	x,y,z
O_4-H_{4o}	O_1	2.502(8)	1.67(-)	163(-)	11(-)	101(-) N_1	x,y,z

a Atoms are labeled in agreement with Figure 1.

b The hydrogen atom involved in the interaction is also indicated.

c The numbers in parentheses are the estimated standard deviations in the last significant digit. Hydrogen atom H_{4o} was included in the structural model at fixed positions obtained from a difference Fourier map. Values listed for distances and angles involving this hydrogen therefore do not include estimated standard deviations.

d All donor atoms belong to the asymmetric unit for which the fractional atomic coordinates are given in Table 2.

CRYSTAL STRUCTURE ANALYSIS REPORT and TABLES for

(2-Oxo-4(E)-hexen-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III) (dichloromethane solvate) (39)

Wake Forest X-Ray Facility Reference Code: **MWE8-0999 / BH199**

For: **Prof. M. Welker**

Single crystals of $[O_2N_2C_4H_7]_2Co(NC_5H_5)(C_6OH_9) \cdot CH_2Cl_2$ are, at 233°K, monoclinic, space group $P2_1/c$ - C_{2h}^5 (No. 14) with $\underline{a}$ = 9.081(1)Å, $\underline{b}$ = 8.267(1)Å, $\underline{c}$ = 33.225(4)Å, ß = 96.01(1)°, V = 2480.5(6)Å^3, and Z = 4 {d_{calcd} = 1.474gcm^{-3}; μ_a(MoK$\bar{\alpha}$) = 0.947mm^{-1}}. A total of 6311 independent reflections having 2θ(MoK$\bar{\alpha}$) < 52.8° (the equivalent of 0.9 limiting CuK$\bar{\alpha}$ sphere) were collected on a computer-controlled Bruker P4 autodiffractometer using ω scans and graphite-monochromated MoK$\bar{\alpha}$ radiation. The structure was solved using "Direct Methods" techniques with the Bruker SHELXTL-PC (vers 5.1) software package. The resulting structural parameters have been refined to convergence {$\underline{R}_1$ (unweighted, based on F) = 0.0417 for 3866 independent reflections having 2θ(MoK$\bar{\alpha}$) < 52.8° and $F^2 > 2\sigma(F^2)$ } {R_1 (unweighted, based on F) = 0.0650 and w$\underline{R}_2$ (weighted, based on F^2) = 0.1055 for all 5081 reflections} using counter-weighted full-matrix least-squares techniques and a structural model which incorporated anisotropic thermal parameters for all nonhydrogen atoms. Hydrogen atoms H_{3o} and H_{4o} were located a difference Fourier map and were refined as independent isotropic atoms. The six methyl groups were refined as rigid rotors with sp^3-

hybridized geometry and a C-H bond length of 0.97Å. The initial orientation for each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation of each methyl group was determined by three rotational parameters. The refined positions for the rigid rotor methyl groups gave C-C-H angles which ranged from 105° to 112°. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp^3- or sp^2-hybridization of the carbon atoms and C-H bond lengths of 0.94 - 0.98Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{3o} and H_{4o} refined to final values of 0.08(1) and 0.08(1)$Å^2$, respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (aromatic or methylene) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

All calculations were performed using the SHELXTL-PC (Version 5.1) interactive software package (G. Sheldrick, Bruker-AXS, Madison, WI).

Table 1. Crystal data and structure refinement for (2-Oxo-4(E)-hexen-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)(dichloromethane)

Empirical formula	$C_{19}H_{28}CoN_5O_5(CH_2Cl_2)$
Formula weight	550.32
Temperature	233(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $P2_1/c$ - C_{2h}^5 (N o. 14)
Unit cell dimensions	a = 9.081(1)Å b = 8.267(1)Å, ß = 96.01(1)° c = 33.225(4)Å
Volume	2480.5(6)Å^3
Z, Calculated density	4, 1.474g/cm^{-1}
Absorption coefficient	0.947 mm^{-1}
F(000)	1144
Crystal size	0.25 x 0.40 x 0.70 mm
Theta range for data collection	2.26 to 26.37°
Limiting indices	$-1 \le h \le 11$, $-10 \le k \le 1$, $-41 \le l \le 41$
Reflections collected / unique	6311 / 5081 [R_{int} = 0.0226]
Completeness to theta = 26.37	100.0 %
Absorption correction	Empirical
Max. and min. transmission	0.8467 and 0.7212
Refinement method	Full-matrix least-squares on F^2
Data / parameters	5081 / 333
Goodness-of-fit on F^2	1.039
Final R indices	
[3866 I>2sigma(I) data]	R_1 = 0.0417, wR_2 = 0.0946
[all 5081 data]	R_1 = 0.0650, wR_2 = 0.1055
Largest diff. peak and hole	0.451 and -0.325e$^-$/Å^3

Table 2. Atomic Coordinates for Atoms in Crystalline (2-Oxo-4(E)-hexen-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)(dichloromethane)[a]

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, Å^2 x 10^3 [c]
Co_1	2125(1)	-937(1)	3623(1)	25(1)
O_1	-3(2)	970(3)	3150(1)	38(1)
O_2	3621(2)	-262(3)	4398(1)	38(1)
O_3	4218(2)	-2927(3)	4102(1)	36(1)
O_4	642(2)	-1635(3)	2841(1)	42(1)
O_5	4332(3)	-1427(3)	2597(1)	56(1)
N_1	909(2)	869(3)	3496(1)	29(1)
N_2	2656(2)	280(3)	4095(1)	29(1)
N_3	3341(2)	-2754(3)	3747(1)	28(1)
N_4	1622(2)	-2147(3)	3148(1)	30(1)
N_5	424(2)	-2009(3)	3899(1)	29(1)
C_1	964(3)	2014(3)	3763(1)	32(1)
C_2	1993(3)	1662(4)	4119(1)	34(1)
C_3	60(4)	3521(4)	3707(1)	49(1)
C_4	2249(4)	2769(5)	4472(1)	51(1)
C_5	3294(3)	-3896(3)	3480(1)	31(1)
C_6	2281(3)	-3531(4)	3120(1)	33(1)
C_7	4160(4)	-5421(4)	3536(1)	45(1)
C_8	2043(4)	-4604(4)	2758(1)	48(1)
C_9	-932(3)	-2167(4)	3700(1)	36(1)
C_{10}	-2115(3)	-2782(4)	3875(1)	47(1)
C_{11}	-1924(4)	-3281(5)	4267(1)	55(1)
C_{12}	-530(4)	-3167(5)	4478(1)	52(1)

Table 2. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, $Å^2 \times 10^3$ [c]
C_{13}	607(3)	-2521(4)	4281(1)	40(1)
C_{14}	5090(3)	211(4)	3537(1)	38(1)
C_{15}	3731(3)	113(3)	3353(1)	29(1)
C_{16}	3335(3)	814(4)	2936(1)	34(1)
C_{17}	3565(3)	-224(4)	2577(1)	41(1)
C_{18}	2817(5)	400(6)	2182(1)	73(1)
C_{19}	6404(4)	966(5)	3369(1)	56(1)
	Solvent of Crystallization[d]			
Cl_1	1565(2)	8800(2)	5455(1)	116(1)
C_{1s}	3316(5)	8545(6)	5314(1)	79(1)
Cl_2	3653(2)	6555(2)	5146(1)	86(1)
$Cl_{2'}$	3600(20)	6760(20)	5421(5)	105(5)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] This is one-third of the trace of the orthogonalized U_{ij} tensor.

[d] A disordered dichloromethane solvent molecule of crystallization is present in the lattice with two alternate positions present for a chlorine atom and the hydrogen atoms. The major (90%) orientation is specified by atoms Cl_1, Cl_2, C_{1s}, H_{1sa} and H_{1sb}. The minor (10%) orientation is specified by atoms Cl_1, $Cl_{2'}$, C_{1s}, H_{1sc} and H_{1sd}.

Table 3. Bond Lengths in Crystalline (2-Oxo-4(E)-hexen-4-yl)(pyridine)bis(dimethylglyoximato)cobalt(III)(dichloromethane)[a]

Type[b]	Length, Å	Type[b]	Length, Å
Co_1-N_1	1.878(2)	Co_1-N_3	1.884(2)
Co_1-N_2	1.881(2)	Co_1-N_4	1.884(2)
Co_1-N_5	2.074(2)	Co_1-C_{15}	1.992(3)
O_1-N_1	1.347(3)	O_3-N_3	1.358(3)
O_2-N_2	1.342(3)	O_4-N_4	1.351(3)
O_5-C_{17}	1.212(4)		
N_1-C_1	1.297(4)	N_3-C_5	1.293(3)
N_2-C_2	1.297(4)	N_4-C_6	1.299(4)
N_5-C_9	1.342(4)	N_5-C_{13}	1.333(4)
C_1-C_2	1.458(4)	C_5-C_6	1.464(4)
C_1-C_3	1.493(4)	C_5-C_7	1.486(4)
C_2-C_4	1.485(4)	C_6-C_8	1.490(4)
C_9-C_{10}	1.371(4)	C_{14}-C_{19}	1.505(4)
C_{10}-C_{11}	1.361(5)	C_{15}-C_{16}	1.509(4)
C_{11}-C_{12}	1.384(5)	C_{16}-C_{17}	1.500(4)
C_{12}-C_{13}	1.385(4)	C_{17}-C_{18}	1.504(5)

Table 3. (continued)

Type[b]	Length, Å	Type[b]	Length, Å
C_{14}-C_{15}	1.321(4)		
O_3-H_{3o}	1.09(5)	O_4-H_{4o}	0.93(4)
	Solvent of Crystallization[c]		
Cl_1-C_{1s}	1.717(5)		
C_{1s}-Cl_2	1.774(6)	C_{1s}-$Cl_{2'}$	1.534(17)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] A disordered dichloromethane solvent molecule of crystallization is present in the lattice with two alternate positions present for a chlorine atom and the hydrogen atoms. The major (90%) orientation is specified by atoms Cl_1, Cl_2, C_{1s}, H_{1sa} and H_{1sb}. The minor (10%) orientation is specified by atoms Cl_1, $Cl_{2'}$, C_{1s}, H_{1sc} and H_{1sd}.

Table 4. Bond Angles in Crystalline (2-Oxo-4(E)-hexen-4-yl) (pyridine)bis(dimethylglyoximato)cobalt(III) (dichloromethane)[a]

Type[b,c]	Angle, (deg)	Type[b,c]	Angle, (deg)
N_1-Co_1-N_2	81.76(10)	N_1-Co_1-N_5	89.55(9)
N_2-Co_1-N_3	98.70(10)	N_2-Co_1-N_5	89.98(9)
N_1-Co_1-N_4	98.48(10)	N_3-Co_1-N_5	90.54(9)
N_3-Co_1-N_4	81.06(10)	N_4-Co_1-N_5	90.92(9)
N_1-Co_1-C_{15}	89.57(11)	C_{15}-Co_1-N_5	178.99(11)
N_2-Co_1-C_{15}	90.39(11)	N_1-Co_1-N_3	179.53(10)
N_3-Co_1-C_{15}	90.33(10)	N_2-Co_1-N_4	179.07(10)
N_4-Co_1-C_{15}	88.71(10)		
C_1-N_1-O_1	121.0(2)	C_5-N_3-O_3	119.5(2)
C_1-N_1-Co_1	116.46(19)	C_5-N_3-Co_1	117.13(19)
O_1-N_1-Co_1	122.47(18)	O_3-N_3-Co_1	123.39(17)
C_2-N_2-O_2	121.2(2)	C_6-N_4-O_4	119.5(2)
C_2-N_2-Co_1	116.30(19)	C_6-N_4-Co_1	117.15(19)
O_2-N_2-Co_1	122.50(18)	O_4-N_4-Co_1	123.35(19)
C_{13}-N_5-Co_1	122.12(19)	C_{13}-N_5-C_9	117.0(3)
C_9-N_5-Co_1	120.81(19)		
N_1-C_1-C_2	112.7(2)	N_3-C_5-C_6	112.5(2)
N_1-C_1-C_3	122.8(3)	N_3-C_5-C_7	123.7(3)
C_2-C_1-C_3	124.5(3)	C_6-C_5-C_7	123.7(3)
N_2-C_2-C_1	112.8(2)	N_4-C_6-C_5	112.1(2)
N_2-C_2-C_4	123.8(3)	N_4-C_6-C_8	123.7(3)
C_1-C_2-C_4	123.4(3)	C_5-C_6-C_8	124.2(3)

Table 4. (continued)

Type[b,c]	Angle, (deg)	Type[b,c]	Angle, (deg)
N_5-C_9-C_{10}	123.1(3)	C_{15}-C_{14}-C_{19}	126.3(3)
C_{11}-C_{10}-C_9	119.5(3)	C_{14}-C_{15}-C_{16}	121.5(3)
C_{10}-C_{11}-C_{12}	118.7(3)	C_{14}-C_{15}-Co_1	120.7(2)
C_{11}-C_{12}-C_{13}	118.4(3)	C_{16}-C_{15}-Co_1	117.78(19)
N_5-C_{13}-C_{12}	123.2(3)	O_5-C_{17}-C_{16}	123.6(3)
		O_5-C_{17}-C_{18}	122.2(3)
C_{17}-C_{16}-C_{15}	118.0(3)	C_{16}-C_{17}-C_{18}	114.1(3)
N_3-O_3-H_{3o}	101(2)	N_4-O_4-H_{4o}	98(3)
	Solvent of Crystallization[c]		
Cl_1-C_{1s}-Cl_2	113.3(3)	$Cl_{2'}$-C_{1s}-Cl_1	101.1(8)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

[c] A disordered dichloromethane solvent molecule of crystallization is present in the lattice with two alternate positions present for a chlorine atom and the hydrogen atoms. The major (90%) orientation is specified by atoms Cl_1, Cl_2, C_{1s}, H_{1sa} and H_{1sb}. The minor (10%) orientation is specified by atoms Cl_1, $Cl_{2'}$, C_{1s}, H_{1sc} and H_{1sd}.

Table 5. Anisotropic Thermal Parameters for Nonhydrogen Atoms in Crystalline (2-Oxo-4(E)-hexen-4-yl)(pyridine) bis(dimethylglyoximato)cobalt(III)(dichloromethane)[a,b]

Atom Type[c]	Anisotropic Thermal Parameters ($Å^2 \times 10^3$)					
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Co_1	24(1)	22(1)	28(1)	-2(1)	4(1)	1(1)
O_1	34(1)	42(1)	38(1)	5(1)	-1(1)	8(1)
O_2	36(1)	44(1)	31(1)	-5(1)	-2(1)	0(1)
O_3	35(1)	36(1)	36(1)	4(1)	-3(1)	6(1)
O_4	44(1)	46(1)	33(1)	-3(1)	-8(1)	5(1)
O_5	65(2)	50(2)	56(2)	-13(1)	19(1)	10(1)
N_1	27(1)	26(1)	35(1)	2(1)	7(1)	3(1)
N_2	27(1)	30(1)	31(1)	-4(1)	6(1)	-3(1)
N_3	25(1)	27(1)	32(1)	1(1)	3(1)	1(1)
N_4	27(1)	31(1)	32(1)	-3(1)	3(1)	-2(1)
N_5	29(1)	25(1)	36(1)	-1(1)	7(1)	0(1)
C_1	30(1)	24(1)	45(2)	-3(1)	14(1)	1(1)
C_2	33(2)	32(2)	39(2)	-10(1)	12(1)	-4(1)
C_3	49(2)	32(2)	68(2)	-2(2)	15(2)	10(2)
C_4	53(2)	47(2)	55(2)	-24(2)	8(2)	4(2)
C_5	29(1)	23(1)	42(2)	-4(1)	11(1)	-1(1)
C_6	32(1)	29(1)	39(2)	-7(1)	8(1)	-3(1)
C_7	43(2)	28(2)	64(2)	-3(2)	9(2)	7(1)
C_8	58(2)	40(2)	46(2)	-17(2)	6(2)	-3(2)
C_9	31(2)	32(2)	46(2)	1(1)	6(1)	1(1)
C_{10}	30(2)	41(2)	70(2)	0(2)	12(2)	-2(1)
C_{11}	44(2)	49(2)	76(3)	1(2)	29(2)	-7(2)
C_{12}	60(2)	57(2)	43(2)	4(2)	21(2)	-9(2)
C_{13}	40(2)	42(2)	40(2)	0(1)	9(1)	-5(1)
C_{14}	35(2)	38(2)	43(2)	5(1)	11(1)	0(1)

Table 5. (continued)

Atom Type[c]	Anisotropic Thermal Parameters ($Å^2$ x 10^3)					
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C_{15}	32(1)	22(1)	35(1)	0(1)	13(1)	1(1)
C_{16}	37(2)	28(2)	38(2)	0(1)	12(1)	1(1)
C_{17}	39(2)	47(2)	38(2)	-6(1)	14(1)	-3(2)
C_{18}	81(3)	100(4)	38(2)	-4(2)	8(2)	20(3)
C_{19}	32(2)	61(2)	76(2)	20(2)	13(2)	-4(2)
	Solvent of Crystallization[d]					
Cl_1	88(1)	130(1)	135(1)	-40(1)	36(1)	-20(1)
C_{1s}	76(3)	92(4)	69(3)	-5(3)	6(2)	-31(3)
Cl_2	96(1)	72(1)	87(1)	5(1)	-5(1)	7(1)
$Cl_{2'}$	128(13)	97(12)	85(9)	31(9)	-15(10)	11(9)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] The form of the anisotropic thermal parameter is: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

[c] Atoms are labeled in agreement with Figure 1.

[d] A disordered dichloromethane solvent molecule of crystallization is present in the lattice with two alternate positions present for a chlorine atom and the hydrogen atoms. The major (90%) orientation is specified by atoms Cl_1, Cl_2, C_{1s}, H_{1sa} and H_{1sb}. The minor (10%) orientation is specified by atoms Cl_1, $Cl_{2'}$, C_{1s}, H_{1sc} and H_{1sd}.

Table 6. Atomic Coordinates for Hydrogen Atoms in Crystalline (2-Oxo-4(E)-hexen4-yl)(pyridine)bis(dimethyl glyoximato)cobalt(III)(dichloromethane)[a]

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z
H_{3o}[c]	3920(40)	-1860(60)	4267(12)
H_{4o}[c]	340(50)	-690(60)	2959(12)
H_{3a}	-355	3560	3426
H_{3b}	662	4475	3770
H_{3c}	-736	3488	3880
H_{4a}	2650	2129	4703
H_{4b}	1324	3260	4530
H_{4c}	2950	3608	4419
H_{7a}	4797	-5422	3789
H_{7b}	4762	-5447	3311
H_{7c}	3522	-6365	3524
H_{8a}	1563	-3955	2539
H_{8b}	1412	-5513	2809
H_{8c}	2981	-5002	2684
H_9	-1079	-1840	3427
H_{10}	-3049	-2859	3725
H_{11}	-2724	-3695	4393
H_{12}	-358	-3520	4747
H_{13}	1553	-2440	4424
H_{14}	5252	-234	3798
H_{16a}	2289	1127	2914
H_{16b}	3912	1808	2916
H_{18a}	2819	-458	1983
H_{18b}	3391	1308	2100
H_{18c}	1807	745	2203

Table 6. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z
H_{19a}	6997	1576	3576
H_{19b}	6082	1673	3144
H_{19c}	6991	90	3275
	Solvent of Crystallization		
H_{1sa}	4040	8803	5544
H_{1sb}	3463	9309	5096
H_{1sc}	3343	8741	5024
H_{1sd}	4033	9253	5468

[a] Hydrogen atoms H_{3o} and H_{4o} were located a difference Fourier map and were refined as independent isotropic atoms. The six methyl groups were refined as rigid rotors with sp^3-hybridized geometry and a C-H bond length of 0.97Å. The initial orientation for each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation of each methyl group was determined by three rotational parameters. The refined positions for the rigid rotor methyl groups gave C-C-H angles which ranged from 105° to 112°. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp^3- or sp^2-hybridization of the carbon atoms and C-H bond lengths of 0.94 - 0.98Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{3o} and H_{4o} refined to final values of 0.08(1) and 0.08(1)Å^2, respectively. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (aromatic or methylene) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

[b] Hydrogen atoms which are covalently bonded to carbon are labeled with the same numerical subscript(s) as their carbon atoms with an additional literal subscript (a, b or c) where necessary to distinguish between hydrogens bonded to the same carbon atom. Solvent hydrogen atoms are labeled with the same numerical and literal subscript as their carbon atom with an additional literal subscript (a, b, c or d) to distinguish between the two disordered (90% / 10%) orientations in the lattice.

[c] The numbers in parentheses are the estimated standard deviations in the last significant digit.

Table 7. Torsion angles [deg] for (2-Oxo-4(E)-hexen-4-yl) (pyridine)bis(dimethylglyoximato)cobalt(III) (dichloromethane)[a]

Type[b]	Angle, (deg)	Type[b]	Angle,(deg)
N_2-Co_1-N_1-C_1	-0.7(2)	N_2-Co_1-N_3-O_3	2.5(2)
N_3-Co_1-N_1-C_1	-169(100)	N_4-Co_1-N_3-O_3	-178.4(2)
N_4-Co_1-N_1-C_1	-179.8(2)	C_{15}-Co_1-N_3-O_3	93.0(2)
C_{15}-Co_1-N_1-C_1	-91.2(2)	N_5-Co_1-N_3-O_3	-87.5(2)
N_5-Co_1-N_1-C_1	89.4(2)	N_1-Co_1-N_4-C_6	179.1(2)
N_2-Co_1-N_1-O_1	-179.2(2)	N_2-Co_1-N_4-C_6	74(7)
N_3-Co_1-N_1-O_1	12(14)	N_3-Co_1-N_4-C_6	-0.8(2)
N_4-Co_1-N_1-O_1	1.7(2)	C_{15}-Co_1-N_4-C_6	89.8(2)
C_{15}-Co_1-N_1-O_1	90.4(2)	N_5-Co_1-N_4-C_6	-91.2(2)
N_5-Co_1-N_1-O_1	-89.1(2)	N_1-Co_1-N_4-O_4	0.1(2)
N_1-Co_1-N_2-C_2	1.0(2)	N_2-Co_1-N_4-O_4	-105(7)
N_3-Co_1-N_2-C_2	-179.1(2)	N_3-Co_1-N_4-O_4	-179.8(2)
N_4-Co_1-N_2-C_2	106(7)	C_{15}-Co_1-N_4-O_4	-89.3(2)
C_{15}-Co_1-N_2-C_2	90.5(2)	N_5-Co_1-N_4-O_4	89.8(2)
N_5-Co_1-N_2-C_2	-88.5(2)	N_1-Co_1-N_5-C_{13}	-124.8(2)
N_1-Co_1-N_2-O_2	178.5(2)	N_2-Co_1-N_5-C_{13}	-43.0(2)
N_3-Co_1-N_2-O_2	-1.6(2)	N_3-Co_1-N_5-C_{13}	55.7(2)
N_4-Co_1-N_2-O_2	-76(7)	N_4-Co_1-N_5-C_{13}	136.7(2)
C_{15}-Co_1-N_2-O_2	-92.0(2)	C_{15}-Co_1-N_5-C_{13}	-155(6)
N_5-Co_1-N_2-O_2	88.9(2)	N_1-Co_1-N_5-C_9	52.9(2)
N_1-Co_1-N_3-C_5	-10(14)	N_2-Co_1-N_5-C_9	134.7(2)
N_2-Co_1-N_3-C_5	-179.1(2)	N_3-Co_1-N_5-C_9	-126.6(2)
N_4-Co_1-N_3-C_5	0.0(2)	N_4-Co_1-N_5-C_9	-45.5(2)
C_{15}-Co_1-N_3-C_5	-88.6(2)	C_{15}-Co_1-N_5-C_9	23(6)
N_5-Co_1-N_3-C_5	90.9(2)	O_1-N_1-C_1-C_2	178.8(2)
N_1-Co_1-N_3-O_3	171(100)	Co_1-N_1-C_1-C_2	0.3(3)

Table 7. (continued)

Type[b]	Angle, (deg)	Type[b]	Angle,(deg)
O_1-N_1-C_1-C_3	-1.3(4)	C_9-C_{10}-C_{11}-C_{12}	-0.7(5)
Co_1-N_1-C_1-C_3	-179.8(2)	C_{10}-C_{11}-C_{12}-C_{13}	1.2(6)
O_2-N_2-C_2-C_1	-178.6(2)	C_9-N_5-C_{13}-C_{12}	-1.1(5)
Co_1-N_2-C_2-C_1	-1.1(3)	Co_1-N_5-C_{13}-C_{12}	176.7(3)
O_2-N_2-C_2-C_4	1.2(4)	C_{11}-C_{12}-C_{13}-N_5	-0.3(5)
Co_1-N_2-C_2-C_4	178.7(2)	C_{19}-C_{14}-C_{15}-C_{16}	-1.2(5)
N_1-C_1-C_2-N_2	0.5(3)	C_{19}-C_{14}-C_{15}-Co_1	179.9(3)
C_3-C_1-C_2-N_2	-179.4(3)	N_1-Co_1-C_{15}-C_{14}	130.5(3)
N_1-C_1-C_2-C_4	-179.3(3)	N_2-Co_1-C_{15}-C_{14}	48.7(3)
C_3-C_1-C_2-C_4	0.7(5)	N_3-Co_1-C_{15}-C_{14}	-50.0(3)
O_3-N_3-C_5-C_6	179.1(2)	N_4-Co_1-C_{15}-C_{14}	-131.0(3)
Co_1-N_3-C_5-C_6	0.6(3)	N_5-Co_1-C_{15}-C_{14}	160(6)
O_3-N_3-C_5-C_7	-0.2(4)	N_1-Co_1-C_{15}-C_{16}	-48.4(2)
Co_1-N_3-C_5-C_7	-178.7(2)	N_2-Co_1-C_{15}-C_{16}	-130.2(2)
O_4-N_4-C_6-C_5	-179.6(2)	N_3-Co_1-C_{15}-C_{16}	131.1(2)
Co_1-N_4-C_6-C_5	1.3(3)	N_4-Co_1-C_{15}-C_{16}	0.1(2)
O_4-N_4-C_6-C_8	1.1(4)	N_5-Co_1-C_{15}-C_{16}	-19(6)
Co_1-N_4-C_6-C_8	-177.9(2)	C_{14}-C_{15}-C_{16}-C_{17}	86.7(4)
N_3-C_5-C_6-N_4	-1.2(3)	C_1-C_{15}-C_{16}-C_{17}	-94.4(3)
C_7-C_5-C_6-N_4	178.1(3)	C_{15}-C_{16}-C_{17}-O_5	-16.7(4)
N_3-C_5-C_6-C_8	178.0(3)	C_{15}-C_{16}-C_{17}-C_{18}	166.2(3)
C_7-C_5-C_6-C_8	-2.7(4)		
C_{13}-N_5-C_9-C_{10}	1.7(4)		
Co_1-N_5-C_9-C_{10}	-176.2(2)		
N_5-C_9-C_{10}-C_{11}	-0.8(5)		

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1.

Table 8. Hydrogen-Bonding Interactions in Crystalline (2-Oxo-4(E)-hexen-4-yl) (pyridine)bis(dimethylglyoximato)cobalt(III) (dichloromethane)[a]

Donor[b] Atom D	Acceptor Atom A	Distance Å D···A[c]	Distance Å H···A[c]	Angle, deg. D-H···A[c]	Angle, deg. H-D···A[c]	Angle, deg. H···A-N[c]	Asymmetric[d] Unit of A
O_3-H_{3o}	O_2	2.496(3)	1.43(5)	166(4)	8(2)	102(2) N_2	x,y,z
O_4-H_{4o}	O_1	2.482(3)	1.56(5)	174(4)	4(3)	99(2) N_1	x,y,z

a Atoms are labeled in agreement with Figure 1.

b The hydrogen atom involved in the interaction is also indicated.

c The numbers in parentheses are the estimated standard deviations in the last significant digit.

d All donor atoms belong to the asymmetric unit for which the fractional atomic coordinates are given in Table 2.

CRYSTAL STRUCTURE ANALYSIS REPORT and TABLES for

(1,3(Z)-butadien-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)(chloroform) (45a)

Wake Forest X-Ray Facility Reference Code: **MWE6-0999 / BH165**

For: **Prof. M. Welker**

Single crystals of $[O_2N_2C_4H_7]_2Co(NC_5H_5)(C_4H_5)(Cl_3CH)$ are, at 188°K, triclinic, space group P1 - C_1^1 (No. 1) with $\underline{a}$ = 8.3124(7)Å, $\underline{b}$ = 11.503(1)Å, $\underline{c}$ = 13.374(1)Å, α = 77.949(2)°, β = 73.239(2)°, γ = 71.805(2)°, V = 1153.31(18)Å^3, and Z = 2 {d_{calcd} = 1.557gcm^{-3}; μ_a(MoK$\bar{\alpha}$) = 1.126 mm^{-1}}. A full hemisphere of diffracted intensities (omega scan width of 0.25) was measured using graphite-monochromated MoK$\bar{\alpha}$ radiation on a Bruker SMART CCD Single Crystal Diffraction System. X-rays were provided by a normal-focus sealed x-ray tube operated at 50kV and 40mA. Lattice constants were determined with the Bruker SAINT software package using peak centers for 1452 reflections. A total of 5792 integrated reflection intensities having 2θ(MoK$\bar{\alpha}$) < 48.2° were produced using the Bruker SAINT program. A total of 4485 of these were independent and gave R_{int} = 0.070. The structure was solved using "Direct Methods" techniques with the Bruker SHELXTL-PC (vers 5.10) software package. All stages of weighted full-matrix least-squares refinement were conducted using F_o^2 data and converged to give $\underline{R}_1$ (unweighted, based on F) = 0.049 for 2207 independent absorption-corrected reflections having 2θ(MoK $\bar{\alpha}$) < 48.2° and I>2σ(I) and w$\underline{R}_2$ (weighted, based on F^2) = 0.082 for all 4485 data.

The structural model incorporated anisotropic thermal parameters for all nonhydrogen atoms and isotropic hydrogen atoms. Atoms N_2, C_{22}, C_{25}, C_{36}, C_{2s} and $C_{2s'}$ were restrained with an effective standard deviation of 0.01 so that their U_{ij} components approximated isotropic behavior. Their corresponding isotropic U's were allowed to

vary. Hydrogen atoms H_{1o}, H_{2o}, H_{23o} and H_{24o} were located from a difference Fourier map and were refined as independent isotropic atoms. The eight methyl groups were refined as rigid rotors with sp^3-hybridized geometry and a C-H bond length of 0.98Å. The initial orientation for each methyl group was determined from difference Fourier positions for the hydrogen atoms. The final orientation for each of these methyl groups was determined by one torsion angle parameter per methyl group. The remaining hydrogen atoms were included in the structure factor calculations as idealized atoms (assuming sp^3- or sp^2-hybridization of the carbon atoms and C-H bond lengths of 0.95 - 1.00Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for H_{1o}, H_{2o} and H_{24o} refined to final values of 0.03(4), 0.10(5) and 0.07(4)$Å^2$, respectively; the isotropic thermal parameter for H_{23o} was fixed at a value 1.2 times the equivalent isotropic thermal parameter of O_{23}. The isotropic thermal parameters of the remaining hydrogen atoms were fixed at values 1.2 (aromatic or methylene) or 1.5 (methyl) times the equivalent isotropic thermal parameters of the carbon atoms to which they are covalently bonded.

The absolute structure was determined by refinement of the Flack x parameter to a value of -0.01(3) where expected values are 0(within 3 esd's) for correct and +1 for inverted absolute structures.

All calculations were performed using the SHELXTL-PC(Vers. 5.10) interactive software package (G. Sheldrick, Bruker AXS, Madison, WI).

Table 1. Crystal data and structure refinement for (1,3(Z)-butadien-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)(chloroform) 45a

Empirical formula	$C_{17}H_{24}CoN_5O_4(Cl_3CH)$
Formula weight	540.71
Temperature	188(2)K
Wavelength	0.71073Å
Crystal system, space group	Triclinic, P1 - C_1^1 (No. 1)
Unit cell dimensions	a = 8.3124(7)Å, á = 77.949(2)°
	b = 11.503(1)Å, ß = 73.239(2)°
	c = 13.374(1)Å, ã = 71.805(2)°
Volume	1153.31(18)Å^3
Z, Calculated density	2, 1.557g/cm^{-1}
Absorption coefficient	1.126 mm^{-1}
F(000)	556
Crystal size	0.04 x 0.12 x 0.18 mm
Theta range for data collection	1.60 to 24.10°
Limiting indices	-7≤h≤9, -13≤k≤13, -15≤l≤13
Reflections collected / unique	5792 / 4485 [R_{int} = 0.0702]
Completeness to theta = 24.10°	98.8 %
Absorption correction	Integration
Max. and min. transmission	0.9678 and 0.8342
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4485 / 39 / 618
Goodness-of-fit on F^2	0.793
Final R indices	
[2207 F_o>4sigma(F_o) data]	R_1 = 0.0494, wR_2 = 0.0666
[all 4485 data]	R_1 = 0.1348, wR_2 = 0.0815
Absolute structure parameter	-0.01(3)
Largest diff. peak and hole	0.312 and -0.314 e$^-$/Å^3

Table 2. Atomic Coordinates for Atoms in Crystalline (1,3(Z)-butadien-4-yl)(pyridine)bis(dimethylglyoximato) cobalt(III)(chloroform)[a]

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, $Å^2 \times 10^3$ [c]
		Molecule 1		
Co_1	-2389(2)	-2973(1)	2500(1)	28(1)
O_1	-1242(12)	-810(8)	2293(7)	39(2)
O_2	-1109(10)	-5373(6)	3534(6)	33(2)
O_3	-3515(10)	-5123(6)	2680(6)	35(2)
O_4	-3576(10)	-569(7)	1382(6)	37(2)
N_1	-1057(14)	-2037(9)	2685(8)	29(3)
N_2	-982(12)	-4194(9)	3298(7)	19(3)
N_3	-3721(13)	-3917(9)	2299(8)	24(3)
N_4	-3723(14)	-1751(9)	1652(8)	31(3)
N_5	-4236(12)	-2426(9)	3845(7)	29(3)
C_1	95(17)	-2548(11)	3262(10)	20(3)
C_2	107(17)	-3886(11)	3639(9)	25(4)
C_3	1131(18)	-1871(12)	3524(10)	45(4)
C_4	1403(16)	-4672(11)	4219(10)	43(4)
C_5	-4791(19)	-3370(14)	1676(12)	37(4)
C_6	-4844(18)	-2074(13)	1328(12)	36(4)
C_7	-5884(16)	-3991(11)	1391(10)	43(4)
C_8	-6022(17)	-1185(11)	681(10)	49(4)
C_9	-4895(15)	-1236(10)	3973(11)	37(4)
C_{10}	-6148(18)	-850(12)	4855(11)	45(4)
C_{11}	-6673(18)	-1666(12)	5643(12)	48(4)
C_{12}	-6019(17)	-2908(12)	5562(10)	50(4)

Table 2. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, $Å^2 \times 10^3$ [c]
C_{13}	-4777(18)	-3261(11)	4615(11)	29(4)
C_{15}	-654(14)	-3687(11)	1304(9)	28(3)
C_{16}	244(17)	-3291(11)	384(10)	29(4)
C_{17}	232(17)	-2003(12)	-47(10)	44(4)
C_{18}	1280(20)	-1783(14)	-979(12)	62(5)
		Molecule 2		
Co_2	2393(2)	2973(1)	-2495(1)	26(1)
O_{21}	2411(11)	616(7)	-2921(6)	31(2)
O_{22}	51(10)	5323(7)	-2959(6)	37(2)
O_{23}	2384(12)	5323(7)	-2132(6)	39(2)
O_{24}	4665(11)	630(7)	-2026(7)	41(3)
N_{21}	1664(14)	1850(9)	-2993(8)	26(3)
N_{22}	464(14)	4086(9)	-2990(7)	33(3)
N_{23}	3170(14)	4093(9)	-2023(8)	26(3)
N_{24}	4237(14)	1862(9)	-2003(8)	25(3)
N_{25}	778(12)	2703(9)	-1023(8)	31(3)
C_{21}	319(17)	2290(12)	-3393(11)	24(4)
C_{22}	-416(17)	3625(12)	-3386(10)	25(4)
C_{23}	-523(17)	1569(13)	-3810(11)	48(5)
C_{24}	-1968(16)	4382(11)	-3806(10)	50(4)
C_{25}	4505(17)	3610(13)	-1595(10)	25(4)
C_{26}	5127(18)	2290(12)	-1583(11)	35(4)
C_{27}	5195(16)	4445(11)	-1168(10)	45(4)
C_{28}	6591(16)	1524(11)	-1076(9)	39(4)
C_{29}	842(16)	1582(10)	-485(10)	33(3)

Table 2. (continued)

Atom Type[b]	Fractional Coordinates 10^4x	10^4y	10^4z	Equivalent Isotropic Thermal Parameter, U, $Å^2 \times 10^3$ [c]
C_{30}	-257(17)	1373(12)	503(11)	40(4)
C_{31}	-1387(19)	2379(14)	950(11)	54(5)
C_{32}	-1446(14)	3525(12)	420(10)	41(4)
C_{33}	-333(17)	3701(13)	-575(10)	32(4)
C_{35}	3784(16)	3448(11)	-3871(9)	34(3)
C_{36}	5014(17)	2839(13)	-4642(11)	35(4)
C_{37}	5794(19)	1511(15)	-4685(12)	59(5)
C_{38}	7017(19)	1050(16)	-5502(12)	82(6)
	Solvent Molecule 1			
Cl_1	3928(6)	2333(4)	3070(4)	73(2)
Cl_2	2871(6)	2872(4)	1100(3)	73(2)
Cl_3	1291(6)	1348(4)	2868(4)	75(2)
C_{1s}	2229(19)	2580(12)	445(12)	68(5)
	Solvent Molecule 2[d]			
Cl_4	5978(14)	7753(9)	7003(10)	75(4)
Cl_5	7442(10)	7059(7)	8810(6)	54(2)
Cl_6	9251(13)	8177(8)	6855(8)	69(3)
C_{2s}	7100(40)	8190(30)	7670(20)	42(10)
$Cl_{4'}$	5629(15)	7589(10)	7833(7)	94(4)
$Cl_{5'}$	8770(13)	8093(8)	7852(8)	83(3)
$Cl_{6'}$	8095(12)	8048(8)	5874(7)	103(3)
$C_{2s'}$	7330(60)	8150(40)	7130(30)	76(13)

[a] The numbers in parentheses are the estimated standard deviations in the last significant digit.

[b] Atoms are labeled in agreement with Figure 1. Atom names for atoms in molecule 2 have been incremented by 20 compared to their counterparts in molecule 1.

[c] This is one-third of the trace of the orthogonalized U_{ij} tensor.

[d] The second chloroform molecule present in the lattice appears to be statistically-disordered over two sites. All atoms for this molecule were included in least-squares refinement cycles with occupancies of 0.50.