

**Table S1.**  
Anisotropic Displacement Coefficients<sup>1</sup> ( $\text{\AA}^2 \times 10^3$ )

<u>Atom</u>	<u>U11</u>	<u>U22</u>	<u>U33</u>	<u>U12</u>	<u>U23</u>	<u>U31</u>
Co1	19(1)	17(1)	43(1)	-1(1)	1(1)	3(1)
Co2	13(1)	35(1)	48(1)	-2(1)	-1(1)	6(1)
Co3	16(1)	21(1)	45(1)	-1(1)	-2(1)	2(1)
O1	18(3)	18(3)	48(4)	3(3)	1(3)	0(3)
O2	27(4)	18(3)	58(5)	1(3)	2(3)	0(3)
O3	57(5)	19(3)	56(5)	4(3)	4(4)	2(3)
O4	43(4)	14(3)	50(5)	4(3)	14(4)	1(3)
O5	15(3)	27(4)	51(5)	-2(3)	4(3)	7(3)
O6	15(3)	30(4)	51(5)	-5(3)	-5(3)	16(3)
O7	26(4)	38(4)	68(6)	1(3)	-19(4)	-18(4)
O8	26(4)	43(5)	81(7)	1(3)	-1(4)	-11(4)
O9	28(4)	27(4)	53(5)	7(3)	11(3)	15(3)
O10	200(17)	116(13)	394(33)	63(11)	120(20)	8(16)
C2	75(10)	18(6)	167(16)	-8(6)	73(10)	-17(8)
C3	45(6)	24(5)	40(7)	-3(4)	12(6)	-3(5)
C4	193(18)	19(6)	53(9)	-7(8)	25(11)	-3(6)
C5	30(6)	30(6)	49(7)	6(4)	4(5)	7(5)
C6	41(7)	48(7)	126(14)	24(6)	12(8)	46(8)
C7	40(7)	45(7)	68(9)	-12(5)	-9(6)	-4(6)
C8	72(11)	107(14)	190(22)	5(9)	-34(13)	-85(15)

<sup>1</sup>The general anisotropic thermal parameter expression is  $\exp(-2\pi^2[h^2a^2U_{11} + \dots + 2klb^*c^*U_{23}])$ .

**Table S2**  
H-Atom Coordinates and Isotropic Displacement Coefficients ( $\text{\AA}^2 \times 10^3$ )

<u>Atom</u>	<u>X</u>	<u>Y</u>	<u>Z</u>	<u>U<sub>eq</sub><sup>1</sup></u>
H1	0.1467	0.5463	0.1175	73(46)
H2a	0.2573	0.3525	-0.1365	158(25)
H2b	0.2186	0.3688	-0.2417	158(25)
H2c	0.1932	0.3668	-0.1177	158(25)
H4a	0.1965	0.7501	0.0829	158(25)
H4b	0.2514	0.7629	0.0139	158(25)
H4c	0.2539	0.7234	0.1228	158(25)
H6a	0.0675	0.6360	-0.2142	158(25)
H6b	0.0719	0.6520	-0.0841	158(25)
H6c	0.0230	0.6123	-0.1260	158(25)
H8a	0.0581	0.3423	-0.2597	158(25)
H8b	0.0228	0.3146	-0.1616	158(25)
H8c	-0.0068	0.3552	-0.2502	158(25)

<sup>1</sup>The equivalent isotropic temperature factor,  $U_{eq}$ , is defined as  $1/3\{\sum_i \sum_j (U_{ij} a_i \cdot a_j \cdot a_i \cdot a_j)\}$  where the summations of i and j range from 1 to 3.

**Table S3.**Atomic Coordinates and Equivalent Isotropic Thermal Parameters ( $\text{\AA}^2 \times 10^3$ )

<u>Atom</u>	<u>X</u>	<u>Y</u>	<u>Z</u>	<u>U<sub>eq</sub><sup>a</sup></u>
Co1	0.2228(1)	0.5520(1)	-0.0254(1)	27(1)
Co2	0	0.5000	0	32(1)
Co3	0.1277(1)	0.4601(1)	0.0024(1)	27(1)
O1	0.1693(2)	0.5211(2)	0.0935(6)	28(2)
O2	0.2048(3)	0.4691(3)	-0.0946(6)	34(2)
O3	0.2189(3)	0.6374(3)	0.0350(7)	44(3)
O4	0.2153(3)	0.6901(3)	-0.1280(7)	36(2)
O5	0.1515(3)	0.5775(3)	-0.1371(6)	31(2)
O6	0.0788(2)	0.5270(3)	-0.0762(6)	32(2)
O7	0.0924(3)	0.4012(3)	-0.1005(7)	44(3)
O8	0.0025(3)	0.4285(3)	-0.0969(8)	50(3)
O9	0.2869(3)	0.4552(3)	-0.1795(7)	36(2)
O10	0.1242(8)	0.6183(7)	0.214(2)	237(13)
C1	0.2407(4)	0.4374(4)	-0.1452(8)	27(2)
C2	0.2264(6)	0.3758(4)	-0.162(2)	87(7)
C3	0.2215(4)	0.6835(4)	-0.018(1)	37(4)
C4	0.2317(8)	0.7344(5)	0.057(1)	88(7)
C5	0.0996(4)	0.5722(4)	-0.115(1)	36(4)
C6	0.0618(5)	0.6224(5)	-0.137(1)	72(6)
C7	0.0413(5)	0.3955(5)	-0.128(1)	51(5)
C8	0.0272(6)	0.3481(7)	-0.207(2)	123(9)

<sup>a</sup>The equivalent isotropic temperature factor,  $U_{eq}$ , is defined as  $1/3 \{ \sum_i \sum_j (U_{ij} a_i^* a_j^* \bar{a}_i \cdot \bar{a}_j) \}$  where the summations of  $i$  and  $j$  range from 1 to 3.

**Table S4.**  
Bond Distances(Å) and Angles(°)

Co1-O1	2.008 (6)	Co1-O2a	2.162 (6)
Co1-O3	2.143 (6)	Co1-O9	2.066 (7)
Co1-O1a	2.018 (7)	Co1-O5	2.211 (6)
Co2-O6	2.161 (6)×2	Co2-O4a	2.163 (7)×2
Co2-O8	2.033 (8)×2	Co3-O1	2.041 (6)
Co3-O2a	2.156 (7)	Co3-O5a	2.164 (6)
Co3-O6	2.164 (6)	Co3-O4a	2.123 (7)
Co3-O7	2.016 (7)	O2-C1	1.28 (1)
O3-C3	1.26 (1)	O9-C1	1.24 (1)
O5-C5	1.26 (1)	O6-C5	1.26 (1)
O4-C3	1.29 (1)	O8-C7	1.26 (1)
O7-C7	1.26 (1)	C1-C2	1.51 (1)
C3-C4	1.51 (2)	C5-C6	1.51 (2)
C7-C8	1.49 (2)		
O1-Co1-O1a	174.4(3)	O1-Co1-O2a	78.4(3)
O1-Co1-O3	95.4(3)	O1-Co1-O9	95.2(3)
O1-Co1-O5	91.0(2)	O1a-Co1-O5	84.3(2)
O1a-Co1-O2a	98.0(3)	O1a-Co1-O3	87.2(3)
O1a-Co1-O9	89.6(3)	O2a-Co1-O3	165.8(3)
O2a-Co1-O9	101.8(2)	O2a-Co1-O5	83.2(2)
O3-Co1-O9	91.3(3)	O3-Co1-O5	84.2(3)
O9-Co1-O5	172.7(2)	O6-Co2-O6a	180.0(1)
O6-Co2-O4	79.2(2)×2	O6-Co2-O8	90.2(3)×2
O6a-Co2-O4	100.8(2)×2	O6a-Co2-O8	89.8(3)×2
O4-Co2-O4a	180.0(1)	O4-Co2-O8	88.6(3)×2
O4a-Co2-O8	91.4(3)×2	O8-Co2-O8a	180.0(1)
O1-Co3-O2	77.8(3)	O1-Co3-O5	85.0(2)
O1-Co3-O6	87.5(2)	O1-Co3-O4	89.3(3)
O1-Co3-O7	174.1(3)	O2-Co3-O5	87.4(2)
O2-Co3-O6	99.3(2)	O2-Co3-O4	167.1(3)
O2-Co3-O7	96.5(3)	O5-Co3-O6	168.6(3)
O5-Co3-O4	91.4(3)	O5-Co3-O7	96.3(3)
O6-Co3-O4	80.0(3)	O6-Co3-O7	92.1(3)
O4-Co3-O7	96.4(3)	Co1-O1-Co1a	120.5(3)
Co1-O1-Co3	102.1(3)	Co1a-O1-Co3	98.6(2)
Co1-O2a-Co3	93.7(3)	Co1-O2a-C1a	125.0(6)
Co3-O2a-C1a	138.1(6)	Co1-O3-C3	131.2(7)
Co1-O9-C1	127.2(6)	Co1-O5-C5	126.8(7)
Co3-O5a-Co1a	89.4(2)	Co3-O5a-C5a	142.4(6)
Co2-O6-Co3	94.3(3)	Co2-O6-C5	137.2(6)
Co3-O6-C5	124.2(6)	Co2-O4a-Co3	95.4(3)

Co2-O4a-C3a	132.8(6)	Co3-O4a-C3a	123.8(6)
Co2-O8-C7	134.2(7)	Co3-O7-C7	128.5(7)
O2-C1-O9	122.4(8)	O2-C1-C2	118.5(9)
O9-C1-C2	119.1(9)	O3-C3-O4	125.8(9)
O3-C3-C4	115(1)	O4-C3-C4	119.5(9)
O5-C5-O6	122.5(9)	O5-C5-C6	117.8(9)
O6-C5-C6	119.7(9)	O8-C7-O7	124(1)
O8-C7-C8	119(1)	O7-C7-C8	117(1)

**Table S5**  
Complete Crystallographic Data for  $\text{Co}_5(\text{OH})_2(\text{OOCCH}_3)_8 \cdot 2\text{H}_2\text{O}$  (1)

Crystal Data

Empirical Formula	$\text{H}_{30}\text{C}_{16}\text{Co}_5\text{O}_{20}$
Formula Weight (g/mol)	837.07
Color and Habit	tan petal
Size ( $\text{mm}^3$ )	0.60 x 0.22 x 0.10
Space Group	Tetragonal, $I4_1/a$ (#88)
Unit Cell Dimensions	
a (Å)	23.693(2)
b (Å)	
c (Å)	11.565(5)
$\alpha$ (°)	
$\beta$ (°)	
$\gamma$ (°)	
Volume (Å <sup>3</sup> )	6492(3)
Z	8
Density, calc (g/cm <sup>3</sup> )	1.713
F(000)	

Data Collection

Unit Cell Reflections	48: $28 < 2\theta < 48^\circ$
Two Theta Range (°)	3.5-50
Scan Range (°)	1.20
Scan Type	$\omega$ -2 $\theta$
Scan Speed	1-15°/min
Reflections Collected	3176
Reflections Unique ( $R_{int}$ )	2865 (0.078)
Octants/Indices Collected	hkl, (28,28,13)
Crystal Decomposition (%)	4
Absorption Coefficient ( $\text{mm}^{-1}$ )	2.576
Absorption Correction	6 psi scans
Min./Max. Transmission	0.583-0.733

Refinement

Final Residuals	$R=0.0596$ $R_w=0.0684$
Goodness of Fit (S)	1.76
Largest $\Delta/\sigma$	0.001
Secondary extinction ( $\times 10^{-7}$ )	na
Reflections Observed ( $F > 4\sigma(F)$ )	1798
Parameters Refined	183
Data to Parameter Ratio	9.83
Max./Min. Difference Peaks ( $e/\text{Å}^3$ )	0.93/-0.87

Nicolet R3m/V diffractometer, graphite monochromated Mo  $K\alpha$  radiation ( $\lambda=0.71073\text{Å}$ ), temperature = 20°C, solved by direct methods (SHELXS-86), refined using full-matrix least squares.

**Table S6**  
Bond Valence Sums<sup>a</sup> for  $\text{Co}_3(\text{OH})_2(\text{OOCCH}_3)_8 \cdot 2\text{H}_2\text{O}$

Co1	2.05(3)
Co2	1.94(3)
Co3	1.98(4)
O1	1.24(2)
O2	1.92(5)
O3	1.74(5)
O4	1.91(5)
O5	1.97(6)
O6	2.01(6)
O7	1.86(6)
O8	1.84(6)
O9	1.91(5)

<sup>a</sup>  $\sum s(\text{M-L}) = \sum \exp[(r_0 - r)/0.37]$ .  $s$ =individual bond valences,  $r$ =bond distance in structure and  $r_0$ =empirically derived M-L single bond distance in angstroms (Co-O=1.692, C-O=1.390). Cation-anion distances are from a table in ref. 6.

Figure S1

