Electronic Supplementary Material (ESI) for Dalton Transactions.

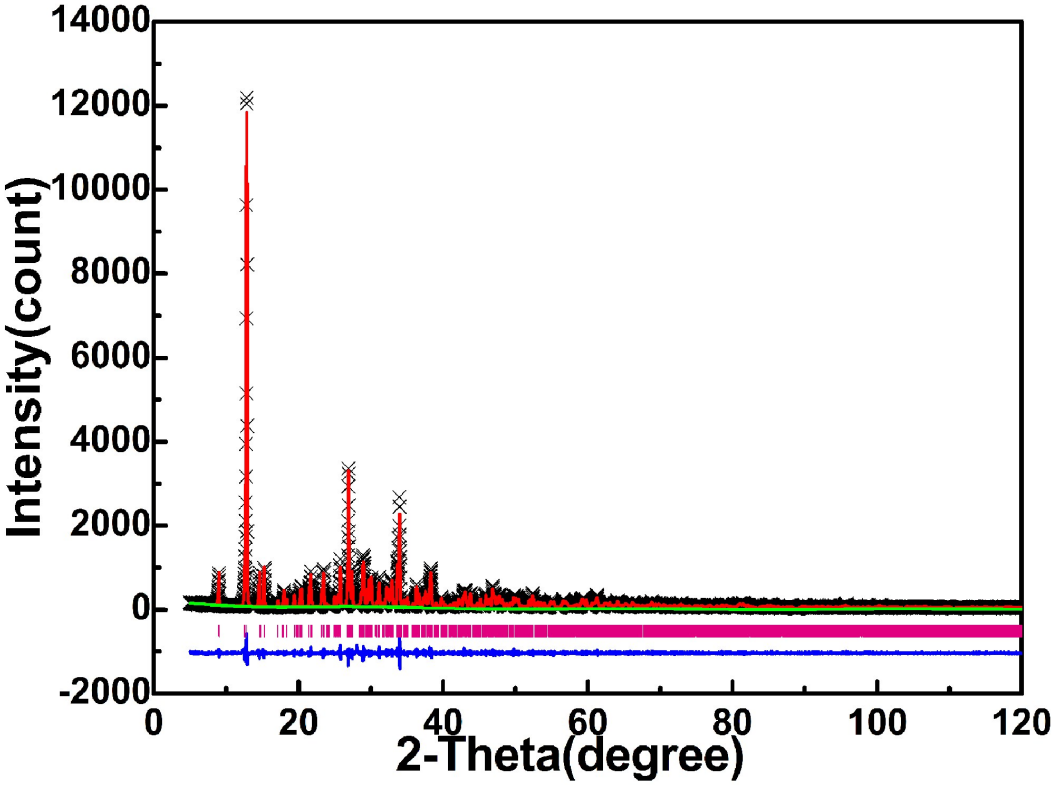

Figure S1. Observed, calculated and difference XRD profiles for **2**. The observed data are indicated by multiplication signs and the profiles calculated by Rietveld refinement and the differences between the observed and calculated patterns are shown by solid lines. The short vertical lines below the profiles mark the positions of all possible Bragg reflections. All peaks were indexed with the lattice parameters refined by the Rietveld analysis: a = 9.735015 Å, b = 12.227146 Å, c = 19.887405 Å, α = 90°, β = 90° and γ = 90°, which agreed with those of single crystal XRD for **1** (Table 1). R_p = 0.0865, R_{wp} = 0.1145 and CHI^2 = 1.884.

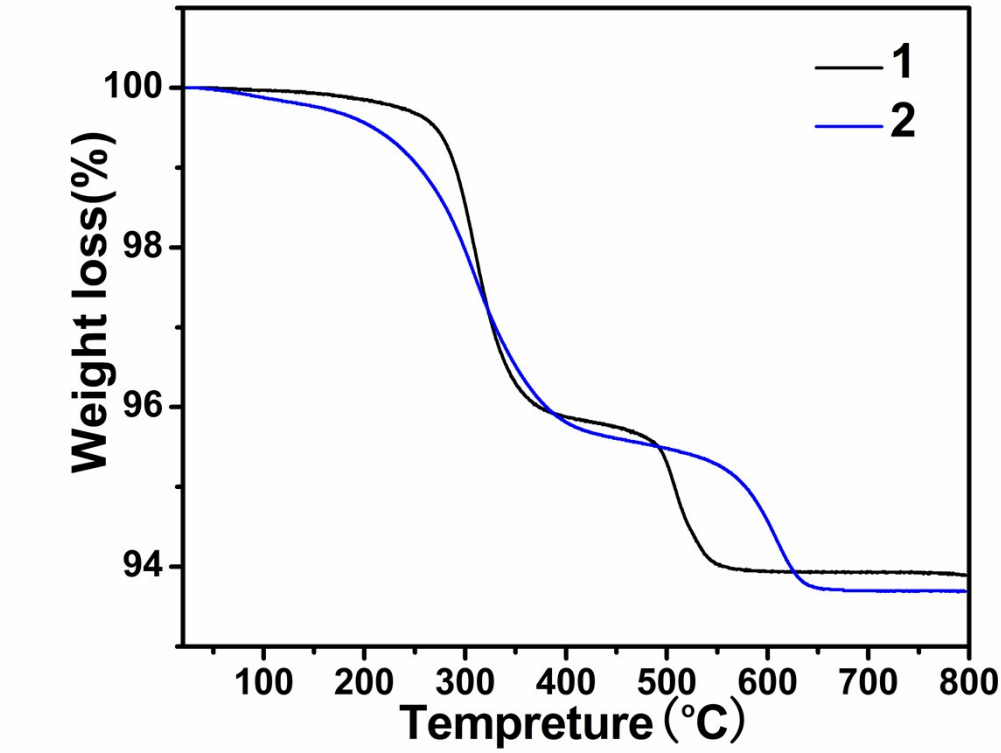

Figure S2. TG curves of **1** and **2**.

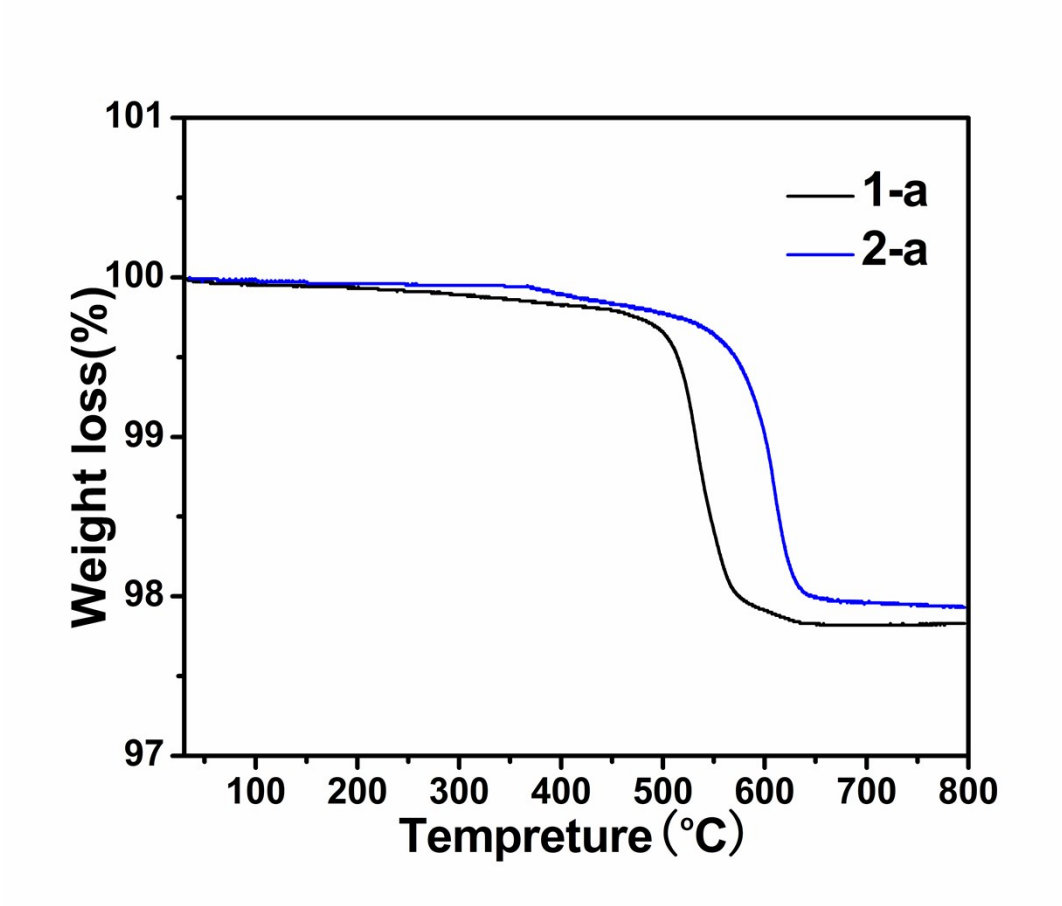

Figure S3. TG curves of **1** heating 10 h at 400 °C (**1-a**) and **2** heating 10 h at 400 °C (**2-a**).

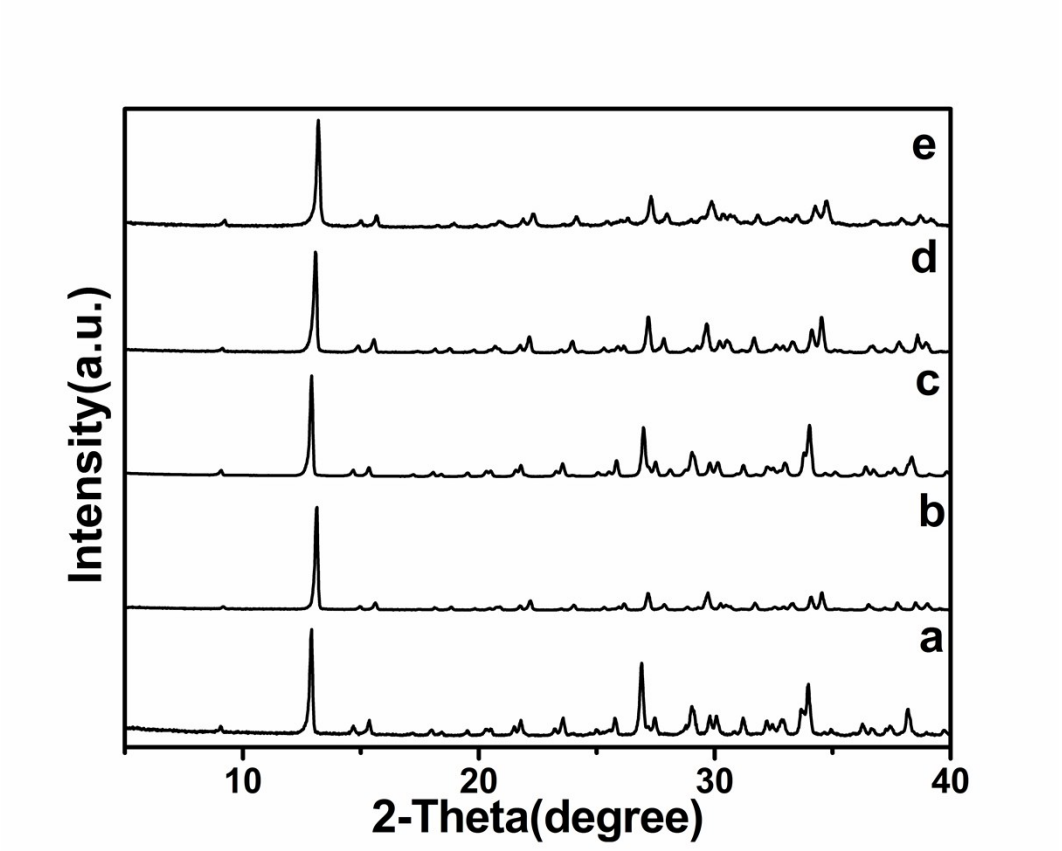

Figure S4. XRD patterns of as-prepared **1** (a), **1** heating 10 h at 400 °C (b), as-prepared **2** (c), **2** heating 10 h at 400 °C (d) and **2** heating 10 h at 500 °C (e).

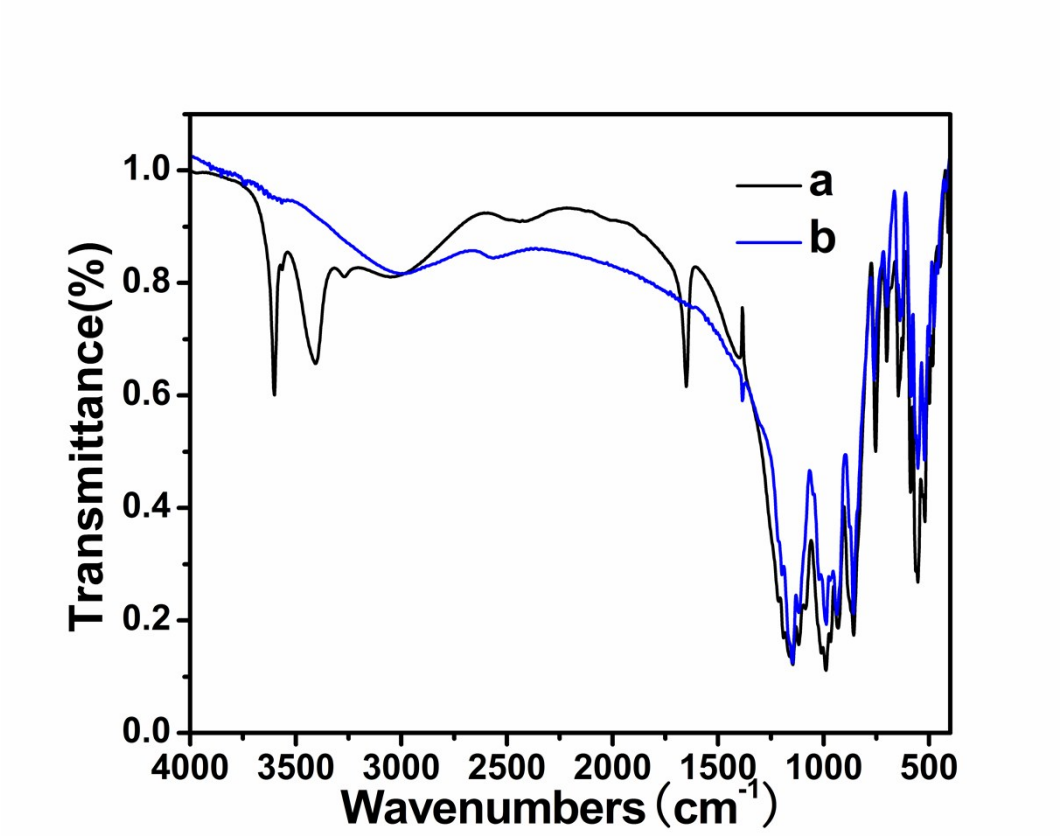

Figure S5. IR spectrum of **1** (a) and **1** heating 10 h at 400 ^{o}C (b)

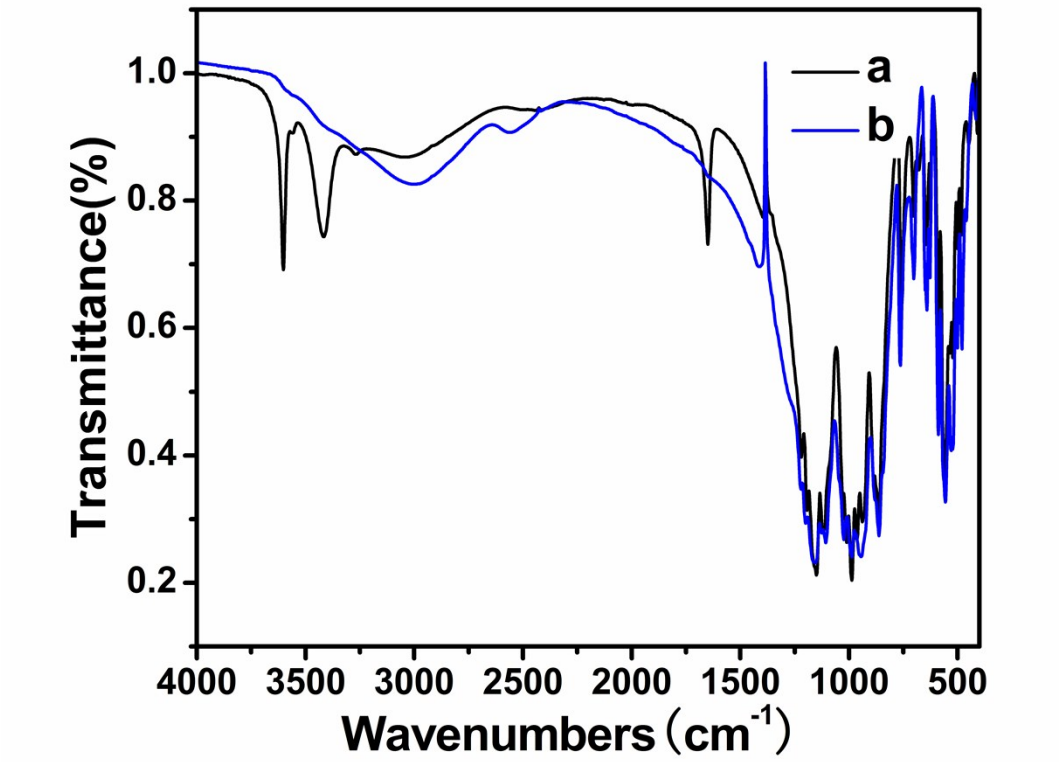

Figure S6. IR spectrum of **2** (a) and **2** heating 10 h at 400 °C (b)

Table S1. Selected bond lengths [Å] and angles [deg] for **1**.

K(1)-O(14)#1	2.604(4)	K(1)-O(1)	2.698(2)
K(1)-O(7)#2	2.802(2)	K(1)-O(9)#3	2.857(2)
K(1)-O(8)#3	2.944(2)	K(1)-O(11)#4	2.974(3)
K(1)-O(2)	2.995(3)	K(1)-O(10)#4	3.292(2)
K(2)-O(14)	2.687(4)	K(2)-O(8)#5	2.701(3)
K(2)-O(13)	2.815(3)	K(2)-O(11)#6	2.856(3)
K(2)-O(4)#5	2.874(3)	K(2)-O(4)	2.953(3)
K(2)-O(12)	2.971(3)	K(2)-O(2)	3.078(3)
Co(1)-O(12)	2.025(2)	Co(1)-O(13)#6	2.081(2)
Co(1)-O(4)#3	2.112(2)	Co(1)-O(8)#3	2.119(2)
Co(1)-O(2)	2.130(2)	Co(1)-O(6)#3	2.158(2)
P(1)-O(4)	1.504(2)	P(1)-O(2)	1.522(2)
P(1)-O(3)	1.554(2)	P(1)-O(1)	1.566(2)
P(2)-O(8)	1.497(2)	P(2)-O(9)	1.547(2)
P(2)-O(5)#8	1.556(2)	P(2)-O(7)	1.561(2)
P(3)-O(12)	1.500(3)	P(3)-O(13)	1.505(2)

P(3)-O(10)	1.562(3)	P(3)-O(11)	1.588(2)
O(1)-B(1)	1.474(4)	O(3)-B(2)	1.459(4)
O(4)-Co(1)#4	2.112(2)	O(4)-K(2)#5	2.874(3)
O(5)-B(1)	1.503(4)	O(5)-P(2)#2	1.556(2)
O(6)-B(1)	1.436(4)	O(6)-B(2)#4	1.449(4)
O(6)-Co(1)#4	2.158(2)	O(7)-B(1)	1.510(4)
O(7)-K(1)#8	2.802(2)	O(8)-Co(1)#4	2.119(2)
O(8)-K(2)#5	2.701(2)	O(8)-K(1)#4	2.944(2)
O(9)-B(2)#9	1.489(4)	O(9)-K(1)#4	2.857(2)
O(10)-B(2)	1.485(4)	O(10)-K(1)#3	3.292(2)
O(11)-K(2)#7	2.856(3)	O(11)-K(1)#3	2.974(3)
O(11)-H(11)	0.816(12)	O(13)-Co(1)#7	2.081(2)
O(14)-K(1)#1	2.604(4)	O(14)-H(14A)	0.818(12)
O(14)-H(14B)	0.8148	B(2)-O(6)#3	1.449(4)
B(2)-O(9)#10	1.489(4)		

O(14)#1-K(1)-O(1)	159.40(11)	O(14)#1-K(1)-O(7)#2	103.20(10)
O(1)-K(1)-O(7)#2	86.19(7)	O(14)#1-K(1)-O(9)#3	105.18(10)
O(1)-K(1)-O(9)#3	92.35(7)	O(7)#2-K(1)-O(9)#3	93.27(7)
O(14)#1-K(1)-O(8)#3	86.11(10)	O(1)-K(1)-O(8)#3	97.06(7)
O(7)#2-K(1)-O(8)#3	144.03(7)	O(9)#3-K(1)-O(8)#3	50.90(6)
O(14)#1-K(1)-O(11)#4	73.06(10)	O(1)-K(1)-O(11)#4	87.13(7)
O(7)#2-K(1)-O(11)#4	102.28(7)	O(9)#3-K(1)-O(11)#4	164.37(7)
O(8)#3-K(1)-O(11)#4	113.64(7)	O(14)#1-K(1)-O(2)	115.12(10)
O(1)-K(1)-O(2)	51.14(7)	O(7)#2-K(1)-O(2)	136.67(7)
O(9)#3-K(1)-O(2)	95.43(7)	O(8)#3-K(1)-O(2)	61.52(7)
O(11)#4-K(1)-O(2)	72.15(7)	O(14)#1-K(1)-O(10)#4	100.75(9)
O(1)-K(1)-O(10)#4	67.26(6)	O(7)#2-K(1)-O(10)#4	61.78(6)
O(9)#3-K(1)-O(10)#4	147.61(7)	O(8)#3-K(1)-O(10)#4	151.34(7)
O(11)#4-K(1)-O(10)#4	44.90(6)	O(2)-K(1)-O(10)#4	90.76(6)
O(14)-K(2)-O(8)#5	102.03(10)	O(14)-K(2)-O(13)	144.49(10)
O(8)#5-K(2)-O(13)	70.99(7)	O(14)-K(2)-O(11)#6	73.87(9)
O(8)#5-K(2)-O(11)#6	123.72(8)	O(13)-K(2)-O(11)#6	139.69(8)
O(14)-K(2)-O(4)#5	84.78(9)	O(8)#5-K(2)-O(4)#5	62.45(7)
O(13)-K(2)-O(4)#5	60.79(7)	O(11)#6-K(2)-O(4)#5	158.52(8)
O(14)-K(2)-O(4)	84.95(10)	O(8)#5-K(2)-O(4)	151.62(8)
O(13)-K(2)-O(4)	87.26(7)	O(11)#6-K(2)-O(4)	84.66(7)
O(4)#5-K(2)-O(4)	91.31(7)	O(14)-K(2)-O(12)	164.28(10)
O(8)#5-K(2)-O(12)	85.91(7)	O(13)-K(2)-O(12)	50.92(7)
O(11)#6-K(2)-O(12)	90.43(7)	O(4)#5-K(2)-O(12)	110.93(7)
O(4)-K(2)-O(12)	94.48(7)	O(14)-K(2)-O(2)	108.84(10)
O(8)#5-K(2)-O(2)	145.02(8)	O(13)-K(2)-O(2)	91.92(7)
O(11)#6-K(2)-O(2)	53.66(7)	O(4)#5-K(2)-O(2)	135.31(7)
O(4)-K(2)-O(2)	49.93(6)	O(12)-K(2)-O(2)	60.33(7)
O(12)-Co(1)-O(13)#6	85.89(10)	O(12)-Co(1)-O(4)#3	88.87(10)

O(13)#6-Co(1)-O(4)#3	86.70(9)	O(12)-Co(1)-O(8)#3	172.58(10)
O(13)#6-Co(1)-O(8)#3	99.44(10)	O(4)#3-Co(1)-O(8)#3	86.29(9)
O(12)-Co(1)-O(2)	94.05(10)	O(13)#6-Co(1)-O(2)	88.74(9)
O(4)#3-Co(1)-O(2)	174.39(9)	O(8)#3-Co(1)-O(2)	91.25(9)
O(12)-Co(1)-O(6)#3	91.24(10)	O(13)#6-Co(1)-O(6)#3	175.64(9)
O(4)#3-Co(1)-O(6)#3	96.53(9)	O(8)#3-Co(1)-O(6)#3	83.72(9)
O(2)-Co(1)-O(6)#3	88.19(9)	O(4)-P(1)-O(2)	114.67(14)
O(4)-P(1)-O(3)	106.96(14)	O(2)-P(1)-O(3)	109.50(13)
O(4)-P(1)-O(1)	110.79(13)	O(2)-P(1)-O(1)	106.33(13)
O(3)-P(1)-O(1)	108.46(13)	O(8)-P(2)-O(9)	110.05(13)
O(8)-P(2)-O(5)#8	111.20(13)	O(9)-P(2)-O(5)#8	105.78(13)
O(8)-P(2)-O(7)	113.09(13)	O(9)-P(2)-O(7)	108.53(13)
O(5)#8-P(2)-O(7)	107.89(13)	O(12)-P(3)-O(13)	112.02(15)
O(12)-P(3)-O(10)	112.30(14)	O(13)-P(3)-O(10)	111.42(14)
O(12)-P(3)-O(11)	111.13(15)	O(13)-P(3)-O(11)	109.44(14)
O(10)-P(3)-O(11)	99.91(13)	B(1)-O(1)-P(1)	129.7(2)
B(2)-O(3)-P(1)	136.7(2)	B(1)-O(5)-P(2)#2	127.5(2)
B(1)-O(6)-B(2)#4	122.6(3)	B(1)-O(7)-P(2)	129.3(2)
B(2)#9-O(9)-P(2)	121.3(2)	B(2)-O(10)-P(3)	124.5(2)
P(3)-O(11)-H(11)	108(3)	K(2)#7-O(11)-H(11)	82(3)
K(1)#3-O(11)-H(11)	140(3)	K(1)#1-O(14)-H(14A)	112(3)
K(2)-O(14)-H(14A)	115(3)	K(1)#1-O(14)-H(14B)	125.5
K(2)-O(14)-H(14B)	111.0	H(14A)-O(14)-H(14B)	87.6
O(6)-B(1)-O(1)	113.8(3)	O(6)-B(1)-O(5)	112.5(3)
O(1)-B(1)-O(5)	103.6(3)	O(6)-B(1)-O(7)	109.9(3)
O(1)-B(1)-O(7)	106.9(3)	O(5)-B(1)-O(7)	109.8(3)
O(6)#3-B(2)-O(3)	113.8(3)	O(6)#3-B(2)-O(10)	111.1(3)
O(3)-B(2)-O(10)	109.0(3)	O(6)#3-B(2)-O(9)#10	111.8(3)
O(3)-B(2)-O(9)#10	104.2(3)	O(10)-B(2)-O(9)#10	106.5(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1 #2 x-1/2,y,-z+3/2

#3 -x+3/2,y+1/2,z #4 -x+3/2,y-1/2,z

#5 -x+2,-y+1,-z+1 #6 x-1/2,-y+3/2,-z+1

#7 x+1/2,-y+3/2,-z+1 #8 x+1/2,y,-z+3/2

#9 -x+2,y-1/2,-z+3/2 #10 -x+2,y+1/2,-z+3/2

Table S2. Selected bond lengths [Å] and angles [deg] for **1-a**.

Co(1)-O(13)	2.011(2)	Co(1)-O(5)	2.071(2)
Co(1)-O(1)	2.088(2)	Co(1)-O(7)	2.089(2)
Co(1)-O(10)	2.150(2)	Co(1)-O(2)	2.194(2)

P(2)-O(10)	1.494(2)	P(2)-O(4)	1.550(2)
P(2)-O(8)	1.556(2)	P(2)-O(12)#4	1.559(2)
K(3)-O(11)	2.711(2)	K(3)-O(4)	2.807(2)
K(3)-O(12)	2.834(2)	K(3)-O(14)	2.917(3)
K(3)-O(1)	2.929(2)	K(3)-O(10)	2.951(2)
K(3)-O(6)	3.259(2)	P(4)-O(13)#6	1.492(3)
P(4)-O(7)#3	1.500(2)	P(4)-O(6)	1.558(2)
P(4)-O(14)	1.598(2)	P(5)-O(5)#6	1.507(2)
P(5)-O(1)	1.522(2)	P(5)-O(9)	1.546(2)
P(5)-O(11)	1.563(2)	O(1)-K(0AB)#3	3.178(6)
O(2)-B(2)	1.434(4)	O(2)-B(15)	1.457(4)
K(0AA)-O(5)#9	2.619(4)	K(0AA)-O(5)#10	2.766(5)
K(0AA)-O(14)	2.845(5)	K(0AA)-O(10)#9	2.900(8)
K(0AA)-O(7)#10	2.914(11)	K(0AA)-O(9)#11	3.257(11)
K(0AA)-O(8)#9	3.370(5)	K(0AA)-O(7)#9	3.386(14)
O(4)-B(15)#13	1.491(4)	O(5)-P(5)#14	1.507(2)
O(5)-K(0AA)#1	2.619(4)	O(5)-K(0AB)#2	2.763(4)
O(5)-K(0AA)#2	2.766(5)	O(5)-K(0AB)#1	2.774(6)
O(6)-B(15)#6	1.485(4)	O(7)-P(4)#3	1.500(2)
O(7)-K(0AB)#1	2.852(5)	O(7)-K(0AA)#2	2.914(11)
O(7)-K(0AA)#1	3.386(14)	O(8)-B(2)#13	1.496(4)
O(8)-K(0AA)#1	3.370(5)	O(9)-B(15)	1.454(4)
O(9)-K(0AA)#8	3.257(11)	O(10)-K(0AB)#1	2.620(4)
O(10)-K(0AA)#1	2.900(8)	O(11)-B(2)#6	1.478(4)
O(12)-B(2)#5	1.506(4)	O(12)-P(2)#5	1.559(2)
O(13)-P(4)#14	1.492(2)	O(13)-K(0AB)#3	3.118(10)
O(14)-K(0AB)	2.672(5)	O(14)-H(14)	0.82(2)
B(15)-O(6)#14	1.485(4)	B(15)-O(4)#15	1.491(4)
B(2)-O(11)#14	1.478(4)	B(2)-O(8)#15	1.497(4)
B(2)-O(12)#4	1.506(4)	K(0AB)-O(10)#9	2.620(4)
K(0AB)-O(5)#10	2.763(4)	K(0AB)-O(5)#9	2.774(6)
K(0AB)-O(7)#9	2.852(5)	K(0AB)-O(13)#3	3.118(10)
K(0AB)-O(1)#3	3.178(6)		

O(13)-Co(1)-O(5)	90.94(10)	O(13)-Co(1)-O(1)	96.64(10)
O(5)-Co(1)-O(1)	172.09(9)	O(13)-Co(1)-O(7)	86.92(10)
O(5)-Co(1)-O(7)	86.16(8)	O(1)-Co(1)-O(7)	91.98(8)
O(13)-Co(1)-O(10)	171.94(9)	O(5)-Co(1)-O(10)	85.41(8)
O(1)-Co(1)-O(10)	87.34(8)	O(7)-Co(1)-O(10)	99.97(9)
O(13)-Co(1)-O(2)	90.07(9)	O(5)-Co(1)-O(2)	95.70(8)
O(1)-Co(1)-O(2)	86.57(8)	O(7)-Co(1)-O(2)	176.49(9)
O(10)-Co(1)-O(2)	83.16(8)	O(10)-P(2)-O(4)	110.26(12)
O(10)-P(2)-O(8)	110.59(12)	O(4)-P(2)-O(8)	106.25(11)
O(10)-P(2)-O(12)#4	113.33(11)	O(4)-P(2)-O(12)#4	108.75(11)
O(8)-P(2)-O(12)#4	107.37(11)	O(11)-K(3)-O(4)	88.71(6)

O(11)-K(3)-O(12)	81.39(6)	O(4)-K(3)-O(12)	89.78(6)
O(11)-K(3)-O(14)	90.20(7)	O(4)-K(3)-O(14)	168.18(7)
O(12)-K(3)-O(14)	101.69(7)	O(11)-K(3)-O(1)	51.51(6)
O(4)-K(3)-O(1)	92.40(6)	O(12)-K(3)-O(1)	132.75(6)
O(14)-K(3)-O(1)	77.77(7)	O(11)-K(3)-O(10)	95.84(6)
O(4)-K(3)-O(10)	51.35(6)	O(12)-K(3)-O(10)	141.13(6)
O(14)-K(3)-O(10)	117.12(7)	O(1)-K(3)-O(10)	59.69(6)
O(11)-K(3)-O(6)	67.44(6)	O(4)-K(3)-O(6)	143.40(6)
O(12)-K(3)-O(6)	60.49(6)	O(14)-K(3)-O(6)	45.30(6)
O(1)-K(3)-O(6)	93.43(6)	O(10)-K(3)-O(6)	152.64(6)
O(13)#6-P(4)-O(7)#3	112.38(15)	O(13)#6-P(4)-O(6)	112.37(13)
O(7)#3-P(4)-O(6)	112.49(13)	O(13)#6-P(4)-O(14)	109.74(16)
O(7)#3-P(4)-O(14)	110.13(14)	O(6)-P(4)-O(14)	98.93(12)
O(5)#6-P(5)-O(1)	113.45(13)	O(5)#6-P(5)-O(9)	106.46(12)
O(1)-P(5)-O(9)	111.65(12)	O(5)#6-P(5)-O(11)	111.25(11)
O(1)-P(5)-O(11)	105.67(12)	O(9)-P(5)-O(11)	108.32(12)
O(5)#9-K(0AA)-O(5)#10	101.14(17)	O(8)#9-K(0AA)-O(7)#9	108.0(2)
O(5)#9-K(0AA)-O(14)	161.7(6)	O(5)#10-K(0AA)-O(14)	82.09(16)
O(5)#9-K(0AA)-O(10)#9	62.25(14)	O(5)#10-K(0AA)-O(10)#9	137.0(6)
O(14)-K(0AA)-O(10)#9	103.1(3)	O(5)#9-K(0AA)-O(7)#10	87.5(2)
O(5)#10-K(0AA)-O(7)#10	59.97(12)	O(14)-K(0AA)-O(7)#10	109.3(2)
O(10)#9-K(0AA)-O(7)#10	145.9(3)	O(5)#9-K(0AA)-O(9)#11	47.68(15)
O(5)#10-K(0AA)-O(9)#11	116.2(2)	O(14)-K(0AA)-O(9)#11	146.3(5)
O(10)#9-K(0AA)-O(9)#11	82.96(12)	O(7)#10-K(0AA)-O(9)#11	64.2(3)
O(5)#9-K(0AA)-O(8)#9	88.61(11)	O(5)#10-K(0AA)-O(8)#9	169.95(14)
O(14)-K(0AA)-O(8)#9	87.89(9)	O(10)#9-K(0AA)-O(8)#9	46.40(7)
O(7)#10-K(0AA)-O(8)#9	123.6(5)	O(9)#11-K(0AA)-O(8)#9	72.25(18)
O(5)#9-K(0AA)-O(7)#9	54.7(2)	O(5)#10-K(0AA)-O(7)#9	76.3(3)
O(14)-K(0AA)-O(7)#9	109.7(4)	O(10)#9-K(0AA)-O(7)#9	61.6(2)
O(7)#10-K(0AA)-O(7)#9	114.70(14)	O(9)#11-K(0AA)-O(7)#9	102.37(12)
B(15)#13-O(4)-P(2)	120.65(17)	B(15)#6-O(6)-P(4)	122.31(19)
B(2)#13-O(8)-P(2)	125.38(18)	B(15)-O(9)-P(5)	135.41(19)
B(2)#6-O(11)-P(5)	127.15(18)	B(2)#5-O(12)-P(2)#5	128.68(18)
P(4)-O(14)-H(14)	112(5)	K(0AB)-O(14)-H(14)	95(5)
K(0AA)-O(14)-H(14)	96(6)	K(3)-O(14)-H(14)	125(5)
O(9)-B(15)-O(2)	112.4(2)	O(9)-B(15)-O(6)#14	108.9(2)
O(2)-B(15)-O(6)#14	111.7(2)	O(9)-B(15)-O(4)#15	105.9(2)
O(2)-B(15)-O(4)#15	110.5(2)	O(6)#14-B(15)-O(4)#15	107.1(2)
O(2)-B(2)-O(11)#14	113.0(2)	O(2)-B(2)-O(8)#15	113.5(2)
O(11)#14-B(2)-O(8)#15	103.8(2)	O(2)-B(2)-O(12)#4	110.2(2)
O(11)#14-B(2)-O(12)#4	107.1(2)	O(8)#15-B(2)-O(12)#4	108.8(2)
O(10)#9-K(0AB)-O(14)	116.4(2)	O(10)#9-K(0AB)-O(5)#10	156.64(12)
O(14)-K(0AB)-O(5)#10	85.36(9)	O(10)#9-K(0AB)-O(5)#9	64.04(9)
O(14)-K(0AB)-O(5)#9	164.3(3)	O(5)#10-K(0AB)-O(5)#9	97.4(2)

O(10)#9-K(0AB)-O(7)#9	72.65(13)	O(14)-K(0AB)-O(7)#9	135.0(4)
O(5)#10-K(0AB)-O(7)#9	86.05(9)	O(5)#9-K(0AB)-O(7)#9	60.65(8)
O(10)#9-K(0AB)-O(13)#3	81.2(2)	O(14)-K(0AB)-O(13)#3	87.4(3)
O(5)#10-K(0AB)-O(13)#3	92.13(17)	O(5)#9-K(0AB)-O(13)#3	107.87(15)
O(7)#9-K(0AB)-O(13)#3	48.98(14)	O(10)#9-K(0AB)-O(1)#3	136.6(4)
O(14)-K(0AB)-O(1)#3	52.70(11)	O(5)#10-K(0AB)-O(1)#3	49.86(8)
O(5)#9-K(0AB)-O(1)#3	138.90(10)	O(7)#9-K(0AB)-O(1)#3	89.32(18)
O(13)#3-K(0AB)-O(1)#3	58.18(16)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2,y-1/2,z #2 x+1/2,-y+1/2,-z+1

#3 -x+1,-y+1,-z+1 #4 -x+1,y-1/2,-z+3/2

#5 -x+1,y+1/2,-z+3/2 #6 -x+3/2,y+1/2,z

#7 x+1/2,-y+3/2,-z+1 #8 x+1,y,z #9 -x+1/2,y+1/2,z

#10 x-1/2,-y+1/2,-z+1 #11 x-1,y,z

#12 -x,-y+1,-z+1 #13 x-1/2,y,-z+3/2

#14 -x+3/2,y-1/2,z #15 x+1/2,y,-z+3/2

#16 x-1/2,-y+3/2,-z+1