Electronic Supplementary Material (ESI) for CrystEngComm.
This journal is © The Royal Society of Chemistry 2018

Solvent-free synthesis and room temperature proton conductivity of new cobalt phosphite-oxalates

Fan Gao[a], Liangliang Huang[a]*, Zhijia Xiu[a], Yanzhen Yin[b], Yike Ma[a], Yanfeng Bi[a]*, Zhiping Zheng[a]

[a]School of Chemistry and Material Science, Liaoning Shihua University, Fushun 113001, Liaoning, P. R. China.

[b]Guangxi Colleges and Universities Key Laboratory of Beibu Gulf Oil and Natural Gas Resource Effective Utilization, Qinzhou University, Qinzhou 535011, China.

* To whom correspondence should be addressed. E-mail: huangll@lnpu.edu.cn (L. L. Huang); biyanfeng@lnpu.edu.cn (Y. F. Bi)

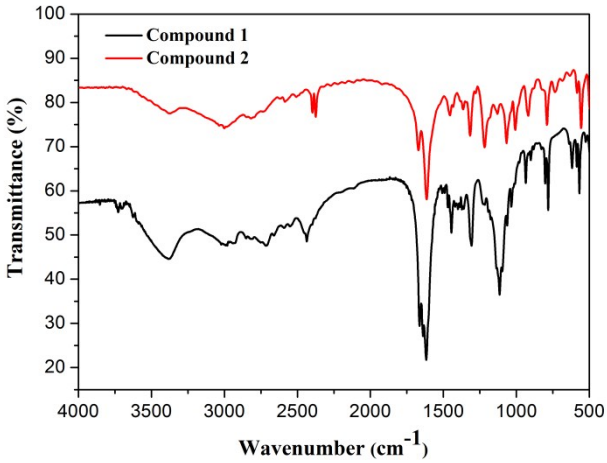

Fig. S1The IR spectra of compounds **1** and **2**.

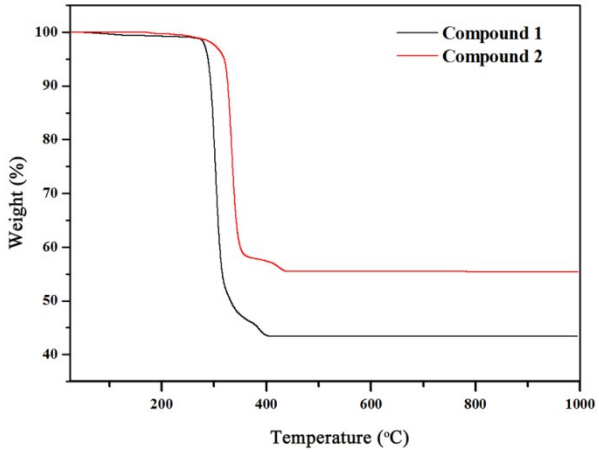

Fig. S2 The TG curve of compounds**1** and **2**.

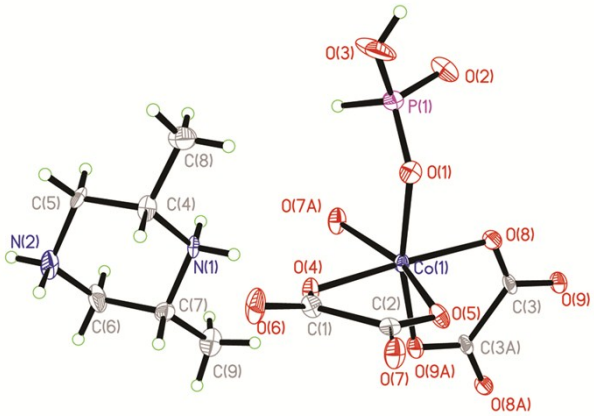

Fig. S3 The asymmetric unit structure of compound **1**.

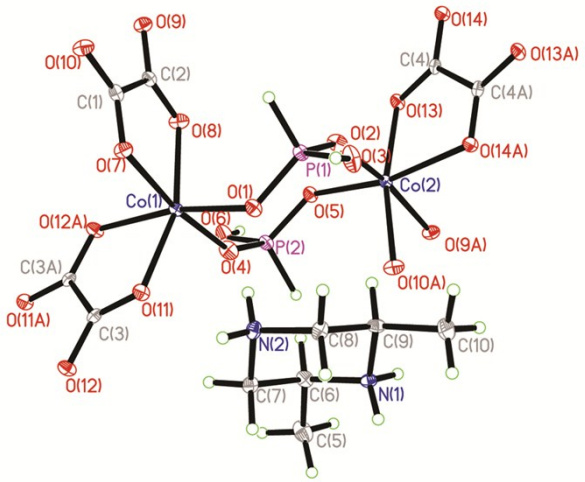

Fig. S4 The asymmetric unit structure of compound **2**.

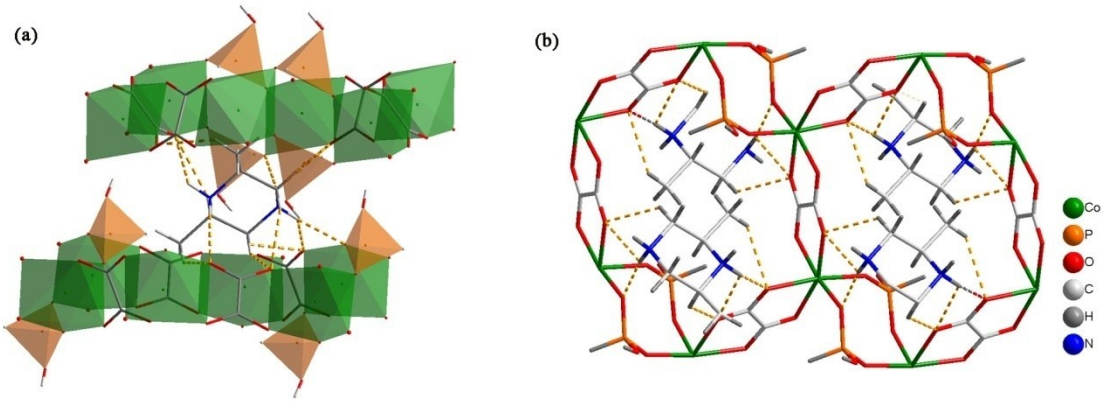

Fig. S5 H-bond interactions between guest molecules and the host framework (a) and (b).

Table S1 Crystal data and structure refinement for compound **1** and **2**

	1	**2**
Empirical formula	$C_9H_{18}CoN_2O_9P$	$C_{10}H_{20}Co_2N_2O_{14}P_2$
Formula weight	388.15	572.08
Temperature	296(2) K	296(2) K
Wavelength	0.71073Å	0.71073 Å
Crystal system. Space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 5.8605(4) Å	a = 15.3007(9) Å
	b = 13.6144(9) Å	b = 8.2832(5) Å
	c= 19.3605(14) Å	c= 16.2601(10) Å
	α = 90°	α = 90°
	β = 105.582(3)°	β = 113.487(2)°
	γ = 90°	γ = 90°
Volume	1487.95(18) $Å^3$	1890.1(2) $Å^3$
Z, Calculated density	4, 1.733g·cm^{-3}	4, 2.010 g·cm^{-3}
Absorption coefficient	1.308 mm^{-1}	2.002 mm^{-1}
F(000)	800	1160
Theta range for data collection	2.6 to 25.1°	2.5 to 28.4°
Refinement method/ unique	25325 / 2621 [R(int)=0.135]	21039 / 4730 [R(int)=0.080]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/ restraints/ parameters	2621 / 0 / 202	4730 / 0 / 273
Goodness-of-fit on F^2	1.058	1.006
Final R indices [I>2sigma(I)]	R1 = 0.0845,	R1 = 0.0377,
	wR2 = 0.1963	wR2 = 0.0810
R indices (all data)	R1 = 0.1058,	R1 = 0.0565,
	wR2 = 0.2057	wR2 = 0.0876
Largest diff. peak and hole	1.39 and -0.97e /$Å^3$	0.59 and -0.93e /$Å^3$

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

Co (1)-O(1)	2.066(6)	Co (1)-O(8)	2.084(6)
Co(1)-O(4)	2.038(6)	Co(1)-O(7)[a]	2.093(7)
Co(1)-O(5)	2.190(7)	Co(1)-O(9)[b]	2.088(7)
P(1)-O(1)	1.499(7)	P(1)-O(3)	1.538(9)
P(1)-O(2)	1.496(8)	P(1)-H(1)	1.3200
O(1)-Co(1)-O(4)	92.2(3)	O(4)-Co(1)-O(7)[a]	82.3(2)
O(1)-Co(1)-O(5)	83.2(3)	O(4)-Co(1)-O(9)[b]	98.6(3)
O(1)-Co(1)-O(8)	88.8(2)	O(5)-Co(1)-O(8)	100.6(2)
O(1)-Co(1)-O(7) [a]	94.1(3)	O(5)-Co(1)-O(7)[a]	158.9(2)
O(1)-Co(1)-O(9) [b]	167.6(2)	O(5)-Co(1)-O(9)[b]	93.4(3)
O(4)-Co(1)-O(5)	76.9(2)	O(7)[a]-Co(1)-O(8)	100.3(2)
O(4)-Co(1)-O(8)	177.1(3)	O(8)-Co(1)-O(9)[b]	80.1(2)
O(1)-P(1)-O(3)	109.3(5)	O(7)[a]-Co(1)-O(9)[b]	93.3(3)
O(1)-P(1)-H(1)	108.00	O(2)-P(1)-O(3)	110.8(5)
O(1)-P(1)-O(2)	115.7(4)	O(2)-P(1)-H(1)	105.00
O(3)-P(1)-H(1)	108.00		

Symmetry transformations used to generate equivalent atoms: [a] -1+x,y,z ; [b] 1-x, 2-y, 2-z

Table S3. Selected bond lengths [Å] and angles [deg.] for compound **2**

Co(1)-O(1)	2.068(2)	Co(2)-O(2)	2.0282(18)
Co(1)-O(4)	2.0127(18)	Co(2)-O(5)	2.067(2)
Co(1)-O(7)	2.1629(19)	Co(2)-O(13)	2.1263(18)
Co(1)-O(8)	2.1209(18)	Co(2)-O(9)[b]	2.1739(18)
Co(1)-O(11)	2.1129(18)	Co(2)-O(10)[b]	2.1033(18)
Co(1)-O(12)[a]	2.170(2)	Co(2)-O(14)[c]	2.179(2)
P(1)-O(1)	1.512(2)	P(2)-O(4)	1.491(2)
P(1)-O(2)	1.4912(19)	P(2)-O(5)	1.514(2)
P(1)-O(3)	1.573(2)	P(2)-O(6)	1.569(3)
P(1)-H(1)	1.2000	P(2)-H(2)	1.2500
O(1)-Co(1)-O(4)	88.94(8)	O(2)-Co(2)-O(5)	91.92(8)
O(1)-Co (1)-O(7)	87.91(8)	O(2)-Co(2)-O(13)	95.26(7)
O(1)-Co(1)-O(8)	102.85(8)	O(2)-Co (2)-O(9)[b]	170.95(7)
O(1)-Co(1)-O(11)	89.65(8)	O(2)-Co(2)-O(10)[b]	93.98(7)
O(1) -Co(1)-O(12)[a]	166.76(7)	O(2)-Co(2)-O(14)[c]	89.07(8)
O(4)-Co(1)-O(7)	171.97(8)	O(5)-Co(2)-O(13)	88.31(8)
O(4)-Co(1)-O(8)	96.94(7)	O(5)-Co(2)-O(9)[c]	88.08(8)
O(4)-Co(1)-O(11)	97.73(7)	O(5)-Co (2)-O(10)[c]	100.42(8)
O(4)-Co(1)-O(12)[a]	92.01(8)	O(5)-Co(2)-O(14)[c]	164.80(7)
O(7)-Co(1)-O(8)	76.59(7)	O(9)[b]-Co(2)-O(13)	93.78(7)
O(7)-Co(1)-O(11)	89.64(7)	O(10)[b]-Co(2)-O(13)	167.07(7)
O(7)- Co (1)-O(12)[a]	92.74(7)	O(13)-Co(2)-O(14)[c]	76.50(7)
O(8)-Co(1)-O(11)	160.86(7)	O(9)[b]-Co(2)-O(10)[b]	77.13(7)
O(8)-Co(1)-O(12)[a]	90.15(8)	O(9)[b]-Co(2)-O(14)[c]	93.31(7)
O(11) -Co(1)-O(12)[a]	77.13(7)	O(10)[b]-Co(2)-O(14)[c]	94.64(8)
O(1)-P(1)-O(2)	118.20(12)	O(4)-P(2)-O(5)	118.05(12)
O(1)-P(1)-O(3)	109.84(11)	O(4)-P(2)-O(6)	107.25(12)
O(2)-P(1)-O(3)	107.99(12)	O(5)-P(2)-O(6)	110.58(12)
O(2)-P(1)-H(1)	107.00	O(6)-P(2)-H(2)	103.00
O(3)-P(1)-H(1)	98.00	O(4)-P(2)-H(2)	107.00
O(1)-P(1)-H(1)	113.00	O(5)-P(2)-H(2)	109.00

Symmetry transformations used to generate equivalent atoms: [b] x, 1+y,z; [a] 1-x, 1-y, 1-z; [c] 2-x,2-y, 2-z

Table S4 Hydrogen bonds for compound **1** [Å and deg.]

D-H…A	d(D-H)	d(H…A)	D(D…A)	<DHA
N(1)-H(1A)…O(6)	0.89	1.82	2.701(11)	170.0
N(1)-H(1B)…O(4)	0.89	1.78	2.658(10)	167.0
N(2)-H(2A)…O(2)	0.89	2.02	2.807(11)	147.0
N(2)-H(2B)…O(1)	0.89	2.00	2.838(10)	157.0
N(2)-H(2B)…O(5)	0.89	2.41	2.993(10)	124.0
O(3)-H(3)…O(2)	0.85	1.69	2.524(11)	168.00
C(5)-H(5A)…O(8)	0.97	2.51	3.171(12)	125.0
C(7)-H(7)…O(3)	0.98	2.38	3.099(14)	129.0
C(8)-H(8B)…O(9)	0.96	2.54	3.319(13)	138.0

Table S5 Hydrogen bonds for compound **2** [Å and deg.]

D-H…A	d(D-H)	d(H…A)	D(D…A)	<DHA
N(1)-H(1A)…O(7)	0.89	1.94	2.777(3)	155.0
N(1)-H(1B)…O(9)	0.89	1.96	2.822(3)	161.0
N(2)-H(2A)…O(1)	0.89	1.95	2.831(3)	170.0
N(2)-H(2A)…O(11)	0.89	2.56	2.970(3)	109.0
N(2)-H(2B)…O(5)	0.89	1.94	2.803(3)	163.0
N(2)-H(2B)…O(13)	0.89	2.46	2.913(3)	112.0
O(3)-H(3A)…O(14)	0.85	1.83	2.674(3)	169.00
O(6)- H(6A)…O(12)	0.85	1.84	2.665(3)	164.00
C(5)-H(5A)…O(7)	0.96	2.53	3.230(4)	130.0
C(7)-H(7A)…O(11)	0.97	2.37	3.048(3)	127.0
C(7)-H(7B)…O(8)	0.97	2.37	3.244(3)	149.0
C(8)-H(8A)…O(8)	0.97	2.50	3.332(3)	144.0
C(8)-H(8B)…O(13)	0.97	2.35	2.986(3)	123.0
C(9)-H(9)…O(10)	0.98	2.50	3.193(3)	127.0
C(10)-H(10A)…O(9)	0.96	2.53	3.262(4)	134.0