

Supporting Information for

Two Novel Organic-Inorganic Hybrid Molybdenum(V) Cobalt/Nickel Phosphate Compounds Based on Isolated Nanosized Mo/Co(Ni)/P Cluster Wheels

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Materials and Instruments

All chemical reagents were commercially available and used without further purification. C, H, N elemental analyses were performed on Perkin-Elmer 2400 CHN elemental analyzer. IR spectra of all compounds were recorded with a Nicolet Impact 410 FTIR spectrometer using pressed KBr pellets in the 4000–450 cm⁻¹ region. TG measurement was carried out on a Diamond thermogravimetric analyzer in flowing N₂ atmosphere from 25 to 800 °C with a heating rate of 10 °C min⁻¹. Solid state fluorescence properties of **1** and **2** were carried out using an F-4600 FL Spectrophotometer. Powder XRD patterns were obtained on Bruker D8X diffractometer equipped with monochromatized Cu-K α (λ = 0.15418 nm) radiation at room temperature, and the data were collected in the range of 5 ° $\leq$ 2 θ $\leq$ 50 °. Magnetic susceptibility measurements were performed on a Quantum Design MPMS SQUID magnetometer using single crystal samples. Two-photon absorption (TPA) cross sections were obtained by an open-aperture Z-scan setup using a Chameleon II femtosecond laser pulse and a Ti:95 sapphire system (680–1080 nm) with a pulse duration of 140 fs and 80 MHz repetition rate.

Table S1. Selected bond lengths (Å) for compounds **1**, **2**.

1			
Mo(1)-O(20)	1.692(4)	Co(2)-O(31)	2.248(4)
Mo(1)-O(1)	1.965(3)	Co(3)-O(4)	2.037(4)
Mo(1)-O(2)	1.968(4)	Co(3)-O(14)	2.072(4)
Mo(1)-O(3)	2.087(4)	Co(3)-N(2)	2.121(5)
Mo(1)-O(12)	2.092(4)	Co(3)-O(17)	2.134(3)
Mo(1)-O(16)	2.206(4)	Co(3)-O(19)	2.152(3)
Mo(1)-Mo(3)	2.6016(8)	Co(3)-O(30)	2.264(3)
Mo(2)-O(26)	1.677(4)	Co(4)-O(5)	2.039(4)
Mo(2)-O(4)	1.963(4)	Co(4)-O(29)#4	2.077(5)
Mo(2)-O(5)	1.966(4)	Co(4)-N(3)	2.107(5)
Mo(2)-O(15)	2.081(4)	Co(4)-O(13)	2.141(3)
Mo(2)-O(9)	2.094(4)	Co(4)-O(23)	2.176(3)
Mo(2)-O(22)	2.173(4)	Co(4)-O(28)	2.240(3)
Mo(2)-Mo(4)	2.6077(6)	Co(5)-O(2)	2.038(3)
Mo(3)-O(25)	1.680(4)	Co(5)-O(14)	2.098(3)
Mo(3)-O(1)	1.942(4)	Co(5)-N(1A)	2.1441(14)
Mo(3)-O(2)	1.953(3)	Co(5)-O(19)	2.150(4)
Mo(3)-O(10)	2.054(4)	Co(5)-O(27)	2.154(4)
Mo(3)-O(11)	2.057(4)	Co(5)-O(32)	2.288(5)
Mo(4)-O(18)	1.670(4)	O(8)-Mo(3)#5	2.454(2)
Mo(4)-O(5)	1.943(4)	O(13)-Co(2)#4	2.140(4)
Mo(4)-O(4)	1.946(3)	O(17)-Co(3)#5	2.134(3)
Mo(4)-O(6)	2.037(4)	O(21)-Mo(4)#5	2.4650(18)
Mo(4)-O(7)	2.047(4)	O(23)-Co(4)#5	2.176(3)
Mo(4)-O(21)	2.4650(18)	O(24)-Co(2)#5	2.155(4)
Co(1)-O(2WB)	1.997(8)	O(27)-Co(5)#5	2.154(4)
Co(1)-O(2WB)#1	1.997(8)	O(28)-Co(4)#5	2.240(3)
Co(1)-O(2WB)#2	1.997(8)	O(29)-Co(4)#4	2.264(3)
Co(1)-O(2WB)#3	1.997(8)	O(31)-Co(2)#5	2.248(4)
Co(1)-O(2W)	2.011(7)	O(32)-Co(5)#5	2.288(5)
Co(1)-O(2W)#2	2.011(7)	O(1W)-O(1W)#1	0.83(7)
Co(1)-O(2W)#3	2.011(7)	O(1W)-O(5WA)	1.17(5)
Co(1)-O(2W)#1	2.011(7)	O(2W)-O(2WB)	1.369(12)
Co(1)-O(2WA)#2	2.027(7)	O(2W)-O(2W)#2	1.67(2)
Co(1)-O(2WA)	2.027(7)	O(5W)-O(1W)#1	0.46(4)
Co(1)-O(2WA)#1	2.027(7)	O(5W)-O(5WA)	1.29(5)
Co(1)-O(2WA)#3	2.027(7)	O(5WA)-O(1W)#1	1.17(5)
Co(2)-O(1)	2.045(3)	O(2WA)-O(2WA)#2	1.52(2)
Co(2)-O(29)	2.107(3)	Co(2)-O(13)#4	2.140(4)
Co(2)-N(1)	2.131(6)	Co(2)-O(24)	2.155(4)

2			
Mo(1)-O(58)	1.680(5)	Ni(6)-N(6)	2.099(5)
Mo(1)-O(9)	1.943(5)	Ni(6)-O(27)#1	2.166(5)
Mo(1)-O(3)	1.969(4)	Ni(6)-O(53)	2.224(4)
Mo(1)-O(21)	2.042(5)	Ni(6)-O(10)	2.294(5)
Mo(1)-O(8)	2.077(5)	Ni(7)-O(1)	2.022(5)
Mo(1)-O(12)	2.401(5)	Ni(7)-N(7)	2.085(6)

Mo(1)-Mo(6)	2.5956(9)	Ni(7)-O(45)#1	2.117(4)
Mo(2)-O(16)	1.686(5)	Ni(7)-O(53)	2.165(5)
Mo(2)-O(2)	1.959(4)	Ni(7)-O(10)	2.212(5)
Mo(2)-O(4)	1.975(5)	Ni(8)-O(28)	2.022(5)
Mo(2)-O(17)	2.053(5)	Ni(8)-O(44)	2.025(4)
Mo(2)-O(5)#1	2.095(5)	Ni(8)-N(2)	2.030(6)
Mo(2)-O(39)	2.196(5)	Ni(8)-O(35)	2.057(4)
Mo(2)-Mo(3)	2.6019(9)	Ni(8)-O(62)	2.099(5)
Mo(3)-O(50)	1.668(5)	Ni(8)-O(15)	2.238(5)
Mo(3)-O(2)	1.953(5)	Ni(9)-O(7W)#2	2.030(5)
Mo(3)-O(4)	1.956(4)	Ni(9)-O(7W)	2.030(5)
Mo(3)-O(22)	2.053(5)	Ni(9)-O(9W)#2	2.044(6)
Mo(3)-O(19)	2.068(4)	Ni(9)-O(9W)	2.044(6)
Mo(3)-O(12)	2.433(5)	Ni(9)-O(8W)	2.051(8)
Mo(4)-O(32)	1.671(5)	Ni(9)-O(8W)#2	2.051(8)
Mo(4)-O(18)	1.952(5)	O(5)-Mo(2)#1	2.095(5)
Mo(4)-O(34)	1.965(5)	O(13)-Ni(4)#1	2.120(5)
Mo(4)-O(48)	2.054(5)	O(27)-Ni(6)#1	2.166(5)
Mo(4)-O(41)#1	2.094(5)	O(37)-Ni(6)#1	2.053(5)
Mo(4)-O(42)	2.150(4)	O(41)-Mo(4)#1	2.094(5)
Mo(4)-Mo(7)	2.6033(9)	O(45)-Ni(7)#1	2.086(5)
Mo(5)-O(63)	1.684(5)	O(4W)-O(4WA)	1.26(3)
Mo(5)-O(28)	1.966(5)	Mo(7)-O(60)	2.024(5)
Mo(5)-O(1)	1.972(4)	Mo(7)-O(54)	2.028(5)
Mo(5)-O(59)	2.080(5)	Mo(7)-O(57)	2.398(4)
Mo(5)-O(38)	2.085(5)	Mo(8)-O(56)	1.674(5)
Mo(5)-O(29)	2.183(5)	Mo(8)-O(1)	1.937(5)
Mo(5)-Mo(8)	2.5986(8)	Mo(8)-O(28)	1.947(4)
Mo(6)-O(51)	1.675(5)	Mo(8)-O(7)	2.043(5)
Mo(6)-O(9)	1.953(5)	Mo(8)-O(40)	2.059(5)
Mo(6)-O(3)	1.975(4)	Mo(8)-O(57)	2.429(5)
Mo(6)-O(52)	2.077(5)	Ni(1)-N(1A)	2.017(6)
Mo(6)-O(20)	2.080(5)	Ni(1)-O(4)	2.034(5)
Mo(6)-O(36)	2.165(4)	Ni(1)-O(6)	2.057(5)
Mo(7)-O(61)	1.672(4)	Ni(1)-O(11)	2.059(5)
Mo(7)-O(34)	1.949(5)	Ni(1)-O(14)	2.078(4)
Mo(7)-O(18)	1.955(5)	Ni(1)-O(15)	2.239(5)
Ni(3)-O(27)	2.053(5)	Ni(1)-Ni(2)	2.8892(13)
Ni(3)-O(23)	2.070(5)	Ni(2)-N(3)	2.038(6)
Ni(3)-N(5)	2.108(6)	Ni(2)-O(3)	2.044(4)
Ni(3)-O(49)	2.324(5)	Ni(2)-O(14)	2.049(5)
Ni(4)-O(2)	1.978(4)	Ni(2)-O(6)	2.053(5)
Ni(4)-O(45)	2.005(4)	Ni(2)-O(35)	2.054(5)
Ni(4)-N(1)	2.095(7)	Ni(2)-O(15)	2.213(4)
Ni(4)-O(23)	2.105(5)	Ni(3)-O(9)	2.022(5)
Ni(4)-O(13)#1	2.120(5)	Ni(3)-O(37)	2.050(5)
Ni(4)-O(49)	2.187(5)	Ni(5)-O(11)	2.039(5)
Ni(5)-O(44)	2.012(5)	Ni(5)-O(62)	2.156(4)
Ni(5)-O(18)	2.025(5)	Ni(5)-O(15)	2.252(4)
Ni(5)-N(4)	2.034(6)	Ni(5)-Ni(8)	2.8861(13)
Ni(6)-O(37)#1	2.053(5)	Ni(6)-O(34)	1.995(5)

Table S2. Selected bond angles (°) for compounds **1**, **2**.

1			
O(2WB)-Co(1)-O(2WB)#1	65.6(7)	N(2)-Co(3)-O(17)	95.02(15)
O(2WB)-Co(1)-O(2WB)#2	114.4(7)	O(14)-Co(3)-O(19)	78.58(15)
O(2WB)#1-Co(1)-O(2WB)#2	180.0(5)	N(2)-Co(3)-O(19)	95.65(17)
O(2WB)-Co(1)-O(2WB)#3	180.0(7)	O(17)-Co(3)-O(19)	155.77(14)
O(2WB)#1-Co(1)-O(2WB)#3	114.4(7)	O(4)-Co(3)-O(30)	86.72(18)
O(2WB)#2-Co(1)-O(2WB)#3	65.6(7)	O(14)-Co(3)-O(30)	101.12(19)
O(2WB)#1-Co(1)-O(2W)	94.0(5)	O(17)-Co(3)-O(30)	72.20(15)
O(2WB)#2-Co(1)-O(2W)	86.0(5)	O(19)-Co(3)-O(30)	88.64(15)
O(2WB)#3-Co(1)-O(2W)	140.0(4)	O(5)-Co(4)-O(29)#4	169.48(18)
O(2WB)-Co(1)-O(2W)#2	86.0(5)	O(5)-Co(4)-N(3)	88.1(2)
O(2WB)#1-Co(1)-O(2W)#2	140.0(4)	O(29)#4-Co(4)-N(3)	84.2(2)
O(2WB)#2-Co(1)-O(2W)#2	40.0(4)	O(5)-Co(4)-O(13)	95.19(15)
O(2WB)#3-Co(1)-O(2W)#2	94.0(5)	O(29)#4-Co(4)-O(13)	78.84(16)
O(2W)-Co(1)-O(2W)#2	49.0(7)	N(3)-Co(4)-O(13)	97.7(2)
O(2WB)-Co(1)-O(2W)#3	140.0(4)	O(5)-Co(4)-O(23)	99.6(2)
O(2WB)#1-Co(1)-O(2W)#3	86.0(5)	O(29)#4-Co(4)-O(23)	88.9(2)
O(2WB)#2-Co(1)-O(2W)#3	94.0(5)	N(3)-Co(4)-O(23)	101.51(18)
O(2WB)#3-Co(1)-O(2W)#3	40.0(4)	O(13)-Co(4)-O(23)	156.05(15)
O(2W)-Co(1)-O(2W)#3	180.0(8)	O(5)-Co(4)-O(28)	88.43(19)
O(2W)#2-Co(1)-O(2W)#3	131.0(7)	O(29)#4-Co(4)-O(28)	99.9(2)
O(2WB)-Co(1)-O(2W)#1	94.0(5)	N(3)-Co(4)-O(28)	173.7(3)
O(2WB)#1-Co(1)-O(2W)#1	40.0(4)	O(13)-Co(4)-O(28)	87.87(15)
O(2WB)#2-Co(1)-O(2W)#1	140.0(4)	O(23)-Co(4)-O(28)	73.93(14)
O(2WB)#3-Co(1)-O(2W)#1	86.0(5)	O(2)-Co(5)-O(14)	169.19(17)
O(2W)-Co(1)-O(2W)#1	131.0(7)	O(2)-Co(5)-N(1A)	88.81(12)
O(2W)#2-Co(1)-O(2W)#1	180.0(6)	O(14)-Co(5)-N(1A)	85.49(12)
O(2W)#3-Co(1)-O(2W)#1	49.0(7)	O(2)-Co(5)-O(19)	93.86(15)
O(2WB)-Co(1)-O(2WA)#2	92.3(5)	O(14)-Co(5)-O(19)	78.06(15)
O(2WB)#1-Co(1)-O(2WA)#2	126.2(4)	N(1A)-Co(5)-O(19)	99.49(12)
O(2WB)#2-Co(1)-O(2WA)#2	53.8(4)	O(2)-Co(5)-O(27)	100.04(16)
O(2WB)#3-Co(1)-O(2WA)#2	87.7(5)	O(14)-Co(5)-O(27)	90.13(17)
O(2W)-Co(1)-O(2WA)#2	97.9(5)	N(1A)-Co(5)-O(27)	101.77(15)
O(2W)#2-Co(1)-O(2WA)#2	80.0(4)	O(19)-Co(5)-O(27)	154.74(18)
O(2W)#3-Co(1)-O(2WA)#2	82.1(5)	O(2)-Co(5)-O(32)	86.40(15)
O(2W)#1-Co(1)-O(2WA)#2	100.0(4)	O(14)-Co(5)-O(32)	100.40(17)
O(2WB)-Co(1)-O(2WA)	53.8(4)	N(1A)-Co(5)-O(32)	171.08(18)
O(2WB)#1-Co(1)-O(2WA)	87.7(5)	O(19)-Co(5)-O(32)	88.3(2)
O(2WB)#2-Co(1)-O(2WA)	92.3(5)	O(27)-Co(5)-O(32)	71.7(2)

O(2WB)#3-Co(1)-O(2WA)	126.2(4)	O(2W)-Co(1)-O(2WA)#3	100.0(4)
O(2W)#2-Co(1)-O(2WA)	97.9(5)	O(24)-Co(2)-O(31)	74.5(2)
O(2W)#3-Co(1)-O(2WA)	100.0(4)	O(4)-Co(3)-O(14)	169.65(17)
O(2WB)-Co(1)-O(2WA)#1	87.7(5)	O(4)-Co(3)-O(17)	98.45(19)
O(2WB)#1-Co(1)-O(2WA)#1	53.8(4)	O(14)-Co(3)-O(17)	90.4(2)
O(2WB)#2-Co(1)-O(2WA)#1	126.2(4)	O(1)-Co(2)-O(29)	168.70(17)
O(2WB)#3-Co(1)-O(2WA)#1	92.3(5)	O(1)-Co(2)-N(1)	88.84(16)
O(2WA)#2-Co(1)-O(2WA)	44.1(7)	O(29)-Co(2)-N(1)	83.90(18)
O(2W)#2-Co(1)-O(2WA)#1	100.0(4)	O(1)-Co(2)-O(13)#4	94.14(14)
O(2W)#3-Co(1)-O(2WA)#1	97.9(5)	O(29)-Co(2)-O(13)#4	78.22(15)
O(2W)#1-Co(1)-O(2WA)#1	80.0(4)	N(1)-Co(2)-O(13)#4	96.9(2)
O(2WA)#2-Co(1)-O(2WA)#1	180.000(1)	O(1)-Co(2)-O(24)	100.13(16)
O(2WA)-Co(1)-O(2WA)#1	135.9(7)	O(29)-Co(2)-O(24)	89.80(18)
O(2WB)-Co(1)-O(2WA)#3	126.2(4)	N(1)-Co(2)-O(24)	101.3(2)
O(2WB)#1-Co(1)-O(2WA)#3	92.3(5)	O(13)#4-Co(2)-O(24)	156.94(18)
O(2WB)#2-Co(1)-O(2WA)#3	87.7(5)	O(1)-Co(2)-O(31)	85.96(14)
O(2WB)#3-Co(1)-O(2WA)#3	53.8(4)	O(29)-Co(2)-O(31)	102.01(17)
O(2W)#2-Co(1)-O(2WA)#3	82.1(5)	N(1)-Co(2)-O(31)	172.6(2)
O(2W)#1-Co(1)-O(2WA)#3	97.9(5)	O(2W)-Co(1)-O(2WA)#1	82.1(5)
O(2WA)#2-Co(1)-O(2WA)#3	135.9(7)	O(2W)#1-Co(1)-O(2WA)	82.1(5)
O(2WA)-Co(1)-O(2WA)#3	180.0(8)	O(2WB)-Co(1)-O(2W)	40.0(4)
O(2WA)#1-Co(1)-O(2WA)#3	44.1(7)	O(2W)-Co(1)-O(2WA)	80.0(4)
O(2W)#3-Co(1)-O(2WA)#3	80.0(4)		

2

N(1A)-Ni(1)-O(4)	93.34(18)	O(9)-Ni(3)-O(27)	97.9(2)
N(1A)-Ni(1)-O(6)	95.76(17)	O(37)-Ni(3)-O(27)	80.92(19)
O(4)-Ni(1)-O(6)	101.05(18)	O(9)-Ni(3)-O(23)	95.72(18)
N(1A)-Ni(1)-O(11)	92.56(17)	O(37)-Ni(3)-O(23)	87.74(18)
O(4)-Ni(1)-O(11)	93.23(18)	O(27)-Ni(3)-O(23)	153.5(2)
O(6)-Ni(1)-O(11)	162.99(19)	O(9)-Ni(3)-N(5)	84.42(17)
N(1A)-Ni(1)-O(14)	174.61(18)	O(37)-Ni(3)-N(5)	89.79(17)
O(4)-Ni(1)-O(14)	91.08(17)	O(27)-Ni(3)-N(5)	96.30(17)
O(6)-Ni(1)-O(14)	80.36(18)	O(23)-Ni(3)-N(5)	107.57(17)
O(11)-Ni(1)-O(14)	90.26(17)	O(9)-Ni(3)-O(49)	96.66(17)
N(1A)-Ni(1)-O(15)	100.62(18)	O(37)-Ni(3)-O(49)	89.23(17)
O(4)-Ni(1)-O(15)	166.04(16)	O(27)-Ni(3)-O(49)	86.57(18)
O(6)-Ni(1)-O(15)	77.59(17)	O(23)-Ni(3)-O(49)	69.34(17)
O(11)-Ni(1)-O(15)	86.32(18)	N(5)-Ni(3)-O(49)	176.79(18)
O(14)-Ni(1)-O(15)	74.97(17)	O(2)-Ni(4)-O(45)	173.1(2)
N(1A)-Ni(1)-Ni(2)	129.54(18)	O(2)-Ni(4)-N(1)	85.32(16)

O(4)-Ni(1)-Ni(2)	120.47(12)	O(45)-Ni(4)-N(1)	88.30(16)
O(6)-Ni(1)-Ni(2)	45.29(13)	O(2)-Ni(4)-O(23)	92.88(18)
O(11)-Ni(1)-Ni(2)	118.90(13)	O(45)-Ni(4)-O(23)	91.53(18)
O(14)-Ni(1)-Ni(2)	45.16(13)	N(1)-Ni(4)-O(23)	107.12(18)
O(15)-Ni(1)-Ni(2)	49.14(10)	O(2)-Ni(4)-O(13)#1	98.70(18)
N(3)-Ni(2)-O(3)	95.76(16)	O(45)-Ni(4)-O(13)#1	79.26(18)
N(3)-Ni(2)-O(14)	170.85(17)	N(1)-Ni(4)-O(13)#1	95.62(19)
O(3)-Ni(2)-O(14)	90.65(17)	O(23)-Ni(4)-O(13)#1	155.25(18)
N(3)-Ni(2)-O(6)	91.22(18)	O(2)-Ni(4)-O(49)	91.40(18)
O(3)-Ni(2)-O(6)	100.75(18)	O(45)-Ni(4)-O(49)	95.04(18)
O(14)-Ni(2)-O(6)	81.15(19)	N(1)-Ni(4)-O(49)	176.40(17)
N(3)-Ni(2)-O(35)	93.63(18)	O(23)-Ni(4)-O(49)	71.50(18)
O(3)-Ni(2)-O(35)	92.62(18)	O(13)#1-Ni(4)-O(49)	86.34(18)
O(14)-Ni(2)-O(35)	92.59(19)	O(44)-Ni(5)-O(18)	97.28(19)
O(6)-Ni(2)-O(35)	165.25(17)	O(44)-Ni(5)-N(4)	92.29(19)
N(3)-Ni(2)-O(15)	97.48(16)	O(18)-Ni(5)-N(4)	91.61(19)
O(3)-Ni(2)-O(15)	166.75(18)	O(44)-Ni(5)-O(11)	163.62(18)
O(14)-Ni(2)-O(15)	76.12(16)	O(18)-Ni(5)-O(11)	96.20(19)
O(6)-Ni(2)-O(15)	78.27(17)	N(4)-Ni(5)-O(11)	96.59(19)
O(35)-Ni(2)-O(15)	87.29(17)	O(44)-Ni(5)-O(62)	83.19(18)
N(3)-Ni(2)-Ni(1)	124.86(17)	O(18)-Ni(5)-O(62)	94.75(18)
O(3)-Ni(2)-Ni(1)	120.43(13)	N(4)-Ni(5)-O(62)	172.61(19)
O(14)-Ni(2)-Ni(1)	45.99(12)	O(11)-Ni(5)-O(62)	86.48(18)
O(6)-Ni(2)-Ni(1)	45.40(13)	O(44)-Ni(5)-O(15)	78.30(18)
O(35)-Ni(2)-Ni(1)	121.71(14)	O(18)-Ni(5)-O(15)	166.35(17)
O(15)-Ni(2)-Ni(1)	49.92(12)	N(4)-Ni(5)-O(15)	101.41(19)
O(9)-Ni(3)-O(37)	173.93(18)	O(11)-Ni(5)-O(15)	86.44(17)
O(44)-Ni(8)-O(35)	164.0(2)	O(62)-Ni(5)-O(15)	72.00(17)
N(2)-Ni(8)-O(35)	94.96(15)	O(44)-Ni(5)-Ni(8)	44.53(12)
O(28)-Ni(8)-O(62)	91.51(19)	O(18)-Ni(5)-Ni(8)	118.47(14)
O(44)-Ni(8)-O(62)	84.33(18)	N(4)-Ni(5)-Ni(8)	126.72(19)
N(2)-Ni(8)-O(62)	176.27(17)	O(11)-Ni(5)-Ni(8)	119.99(12)
O(35)-Ni(8)-O(62)	86.41(18)	O(62)-Ni(5)-Ni(8)	46.46(13)
O(28)-Ni(8)-O(15)	163.73(18)	O(15)-Ni(5)-Ni(8)	49.80(12)
O(44)-Ni(8)-O(15)	78.36(17)	O(34)-Ni(6)-O(37)#1	170.8(2)
N(2)-Ni(8)-O(15)	103.28(17)	O(34)-Ni(6)-N(6)	88.62(18)
O(35)-Ni(8)-O(15)	86.53(17)	O(37)#1-Ni(6)-N(6)	87.67(18)
O(62)-Ni(8)-O(15)	73.31(18)	O(34)-Ni(6)-O(27)#1	93.6(2)
O(28)-Ni(8)-Ni(5)	115.00(13)	O(37)#1-Ni(6)-O(27)#1	78.22(19)
O(44)-Ni(8)-Ni(5)	44.17(14)	N(6)-Ni(6)-O(27)#1	92.99(19)
N(2)-Ni(8)-Ni(5)	128.53(16)	O(34)-Ni(6)-O(53)	87.24(18)
O(35)-Ni(8)-Ni(5)	120.99(14)	O(37)#1-Ni(6)-O(53)	97.37(17)
O(62)-Ni(8)-Ni(5)	48.11(12)	N(6)-Ni(6)-O(53)	172.20(19)
O(15)-Ni(8)-Ni(5)	50.21(11)	O(27)#1-Ni(6)-O(53)	93.86(17)

O(1)-Ni(7)-O(10)	100.99(18)	O(34)-Ni(6)-O(10)	100.6(2)
N(7)-Ni(7)-O(10)	102.58(16)	O(37)#1-Ni(6)-O(10)	88.47(19)
O(45)#1-Ni(7)-O(10)	88.84(18)	N(6)-Ni(6)-O(10)	103.40(19)
O(13)-Ni(7)-O(10)	158.19(19)	O(27)#1-Ni(6)-O(10)	158.48(17)
O(53)-Ni(7)-O(10)	73.58(18)	O(53)-Ni(6)-O(10)	70.93(17)
O(28)-Ni(8)-O(44)	94.64(18)	O(1)-Ni(7)-N(7)	86.0(2)
O(28)-Ni(8)-N(2)	91.72(18)	O(1)-Ni(7)-O(45)#1	169.55(18)
O(44)-Ni(8)-N(2)	93.53(15)	N(7)-Ni(7)-O(45)#1	88.4(2)
O(28)-Ni(8)-O(35)	98.59(18)	O(1)-Ni(7)-O(13)	94.03(17)
N(7)-Ni(7)-O(53)	172.35(19)	N(7)-Ni(7)-O(13)	94.11(16)
O(45)#1-Ni(7)-O(53)	97.99(19)	O(45)#1-Ni(7)-O(13)	77.55(17)
O(13)-Ni(7)-O(53)	91.32(17)	O(1)-Ni(7)-O(53)	88.27(19)

Table S3. Hydrogen bonds for compound **1**, **2**

1				
D-H...A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	∠D-H-A/°
C5B-H5BA...O2	0.93	2.55	3.022(5)	112
C5B-H5BA...O25	0.93	2.36	3.275(4)	170
C5-H5A...O35A	0.93	2.45	3.298(11)	152
C1B-H1BA...N2	0.93	2.58	3.472(6)	161
C6-H6A...O4	0.93	2.58	3.047(10)	111
C10-H10A...O33	0.93	2.49	3.346(13)	154
C11-H11A...O35	0.93	2.29	3.178(12)	160
C11-H11A...O4W	0.93	2.57	3.406(10)	149
C13-H13A...O26	0.93	2.41	3.324(10)	168

2				
D-H...A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	∠D-H-A/°
N2A-H2AB...O24	0.86	2.04	2.890(8)	170
N4A-H4AA...O43	0.86	1.85	2.701(9)	173
C1-H1A...O2	0.93	2.55	2.952(10)	106
C1-H1A...O55	0.93	2.58	3.247(12)	129
C3A-H3AA...O31	0.93	2.32	3.204(10)	159
C5-H5A...O24	0.93	2.40	3.280(11)	157
C6A-H6AA...O17	0.93	2.54	3.205(11)	129
C6A-H6AA...O48	0.93	2.47	3.186(11)	134
C9-H9A...O19	0.93	2.57	3.272(11)	133
C10-H10A...O46	0.93	2.48	3.322(12)	150
C11-H11A...O6	0.93	2.47	3.011(12)	117
C13-H13A...O50	0.93	2.39	3.176(14)	141
C14-H14A...O2W	0.93	2.23	3.121(19)	161

C21-H21A...O24	0.93	2.36	3.256(10)	161
C25-H25A...O9	0.93	2.46	2.880(10)	108
C26-H26A...O34	0.93	2.35	2.913(12)	118
C29-H29A...O50	0.93	2.52	3.086(10)	119
C30-H30A...O37	0.93	2.58	2.984(11)	106
C30-H30A...O47	0.93	2.43	3.278(12)	151
C32-H32A...O32	0.93	2.39	3.136(11)	137
C32-H32A...O61	0.93	2.48	3.266(12)	142
C35-H35A...O1	0.93	2.59	2.932(18)	102
C35A-H35B...O13	0.93	2.46	3.051(11)	121
C37-H37A...O8	0.93	2.20	3.02(2)	146
C37-H37A...O58	0.93	2.50	3.135(19)	126
C39-H39A...O26	0.96	2.30	3.244(16)	170

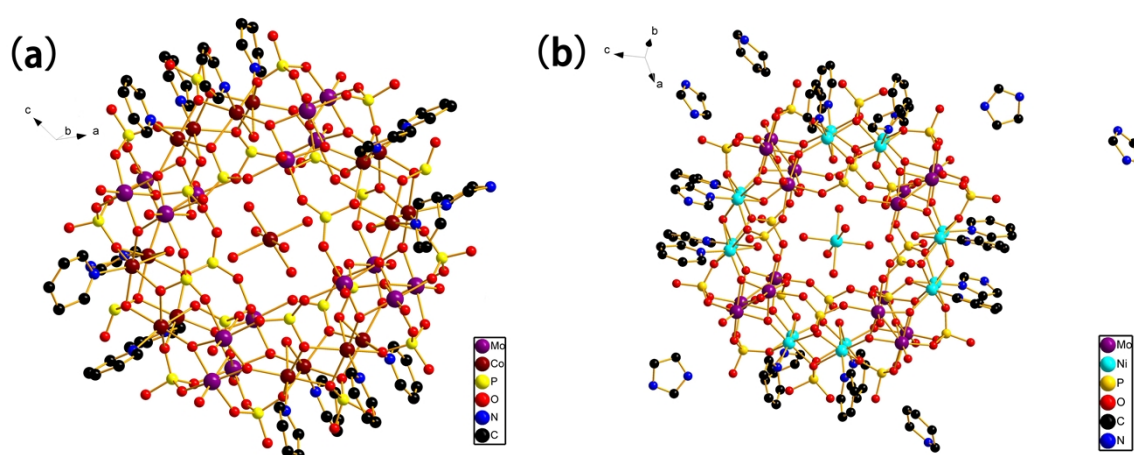


Figure S1. Ball-and-stick representation of the hybrid wheel-shaped cluster of **1** (a) and **2** (b). The H atoms and lattice water molecules are omitted for clarity.

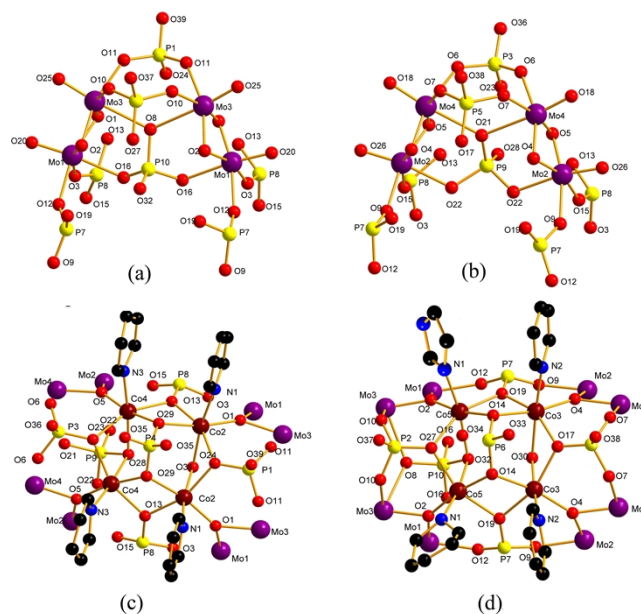


Figure S2. Ball-and-stick representation of four basic building blocks in the wheels of **1**: (a) and (b) two types of $\{\text{Mo}_4\}$ units; (c) and (d) two types of $\{\text{Co}_4\}$ units.

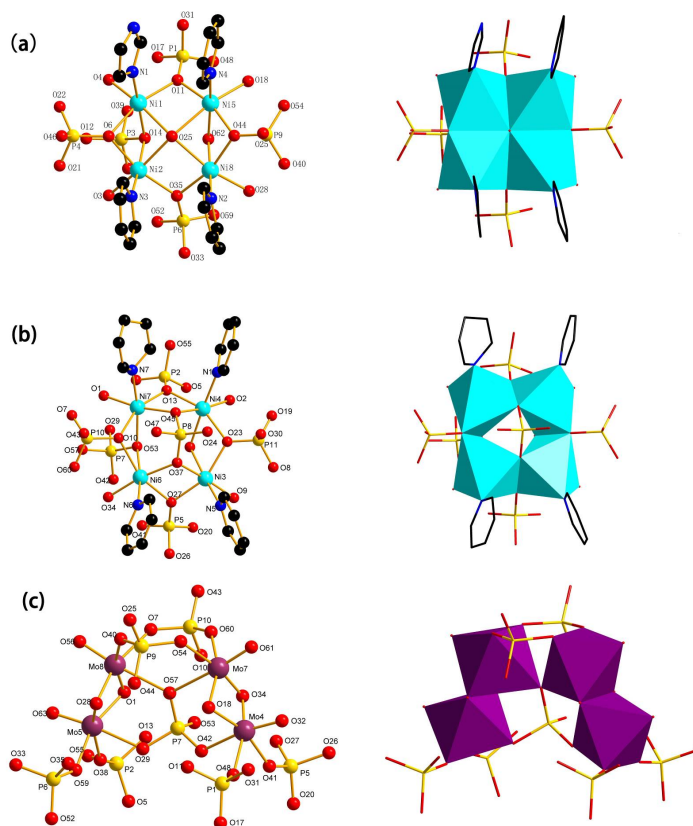


Figure S3. (a) Coordination environment of $\{\text{Ni}_4\text{P}_5\text{N}_4\}$ tetramer in **2**. (b) Coordination environment of $\{\text{Ni}_4\text{P}_6\text{N}_4\}$ tetramer in **2**. (c) Coordination environment of $\{\text{Mo}_4\}$ tetramer in **2**.

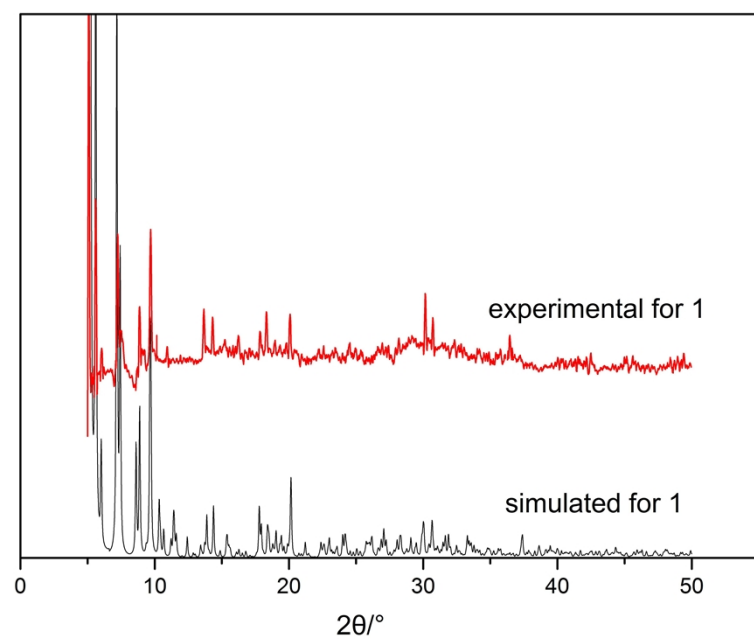


Figure S4. Experimental and simulated power XRD patterns of compound **1**.

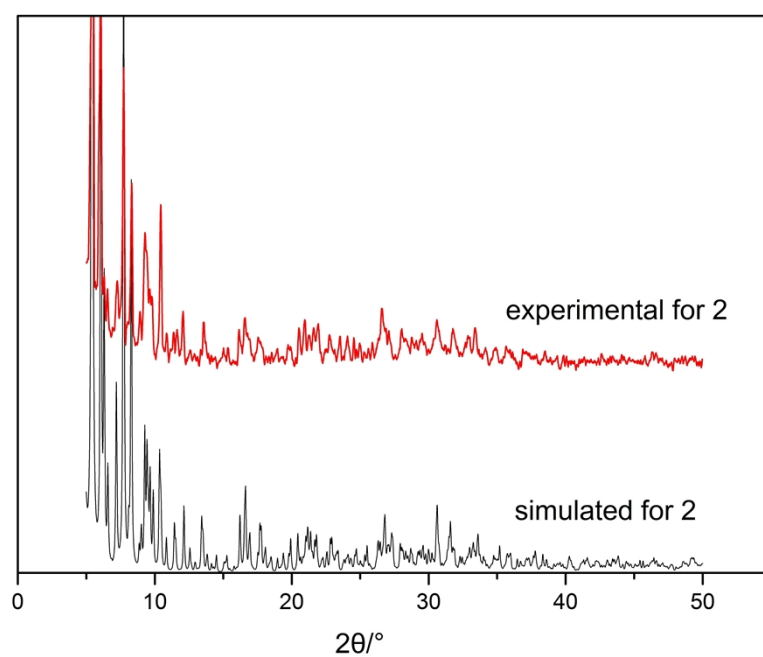


Figure S5. Experimental and simulated power XRD patterns of compound **2**.

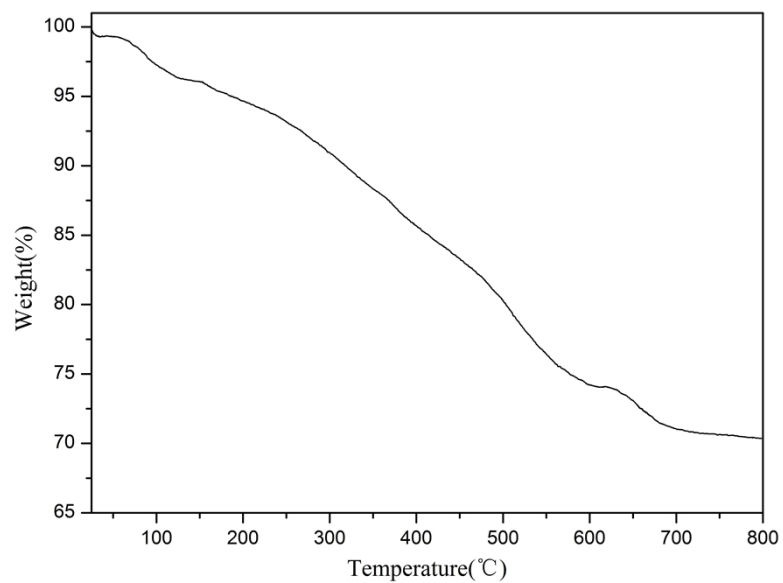


Figure S6. The TG curve of compound 1.

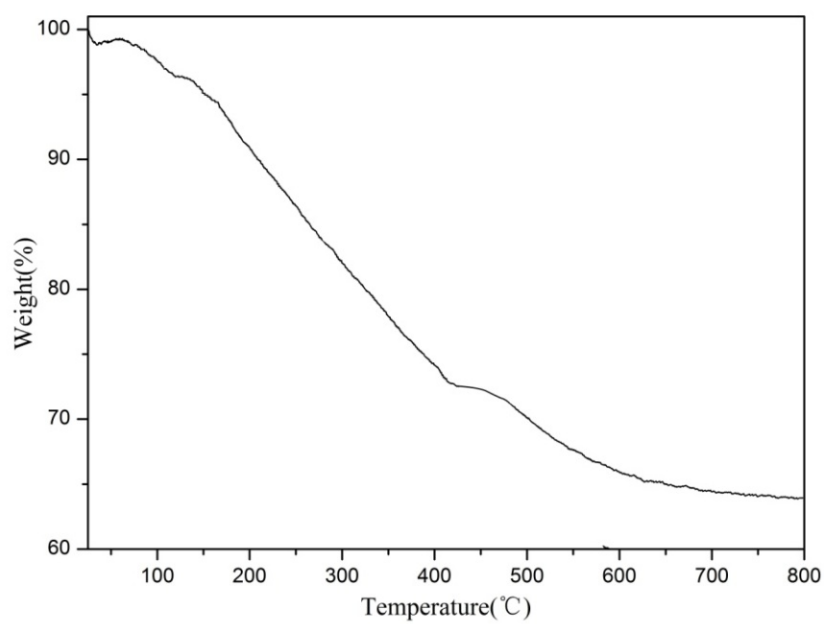


Figure S7. The TG curve of compound 2.

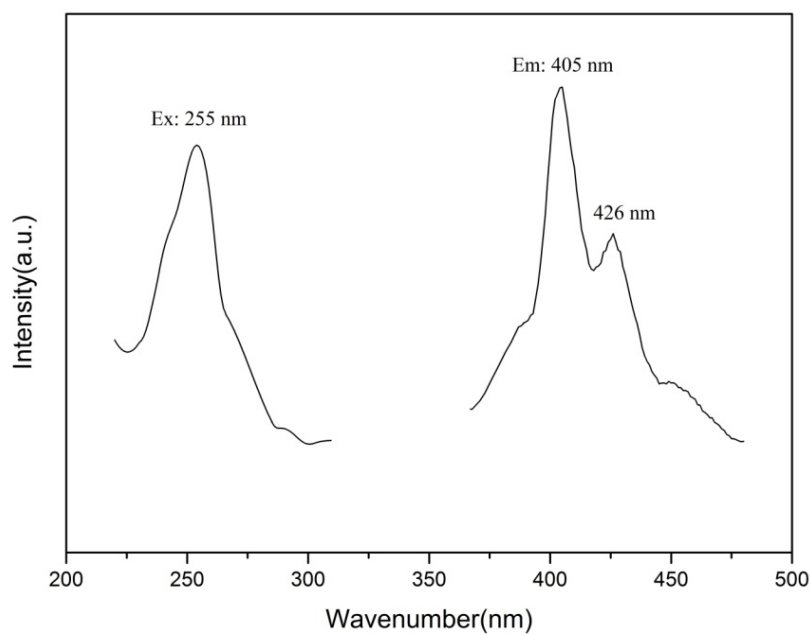


Figure S8. The excitation and emission spectra of compound **1** in the solid state at room temperature.

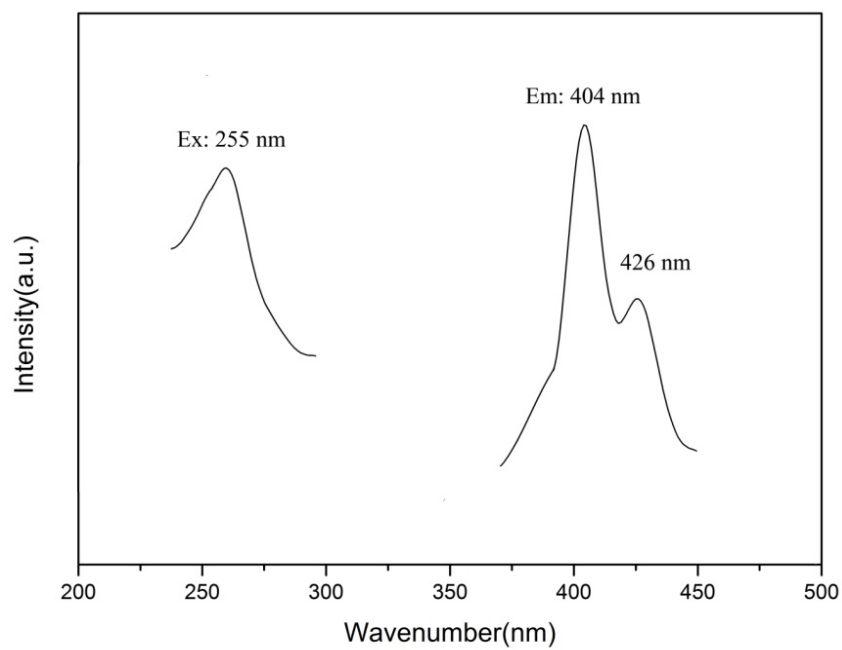


Figure S9. The excitation and emission spectra of compound **2** in the solid state at room temperature.

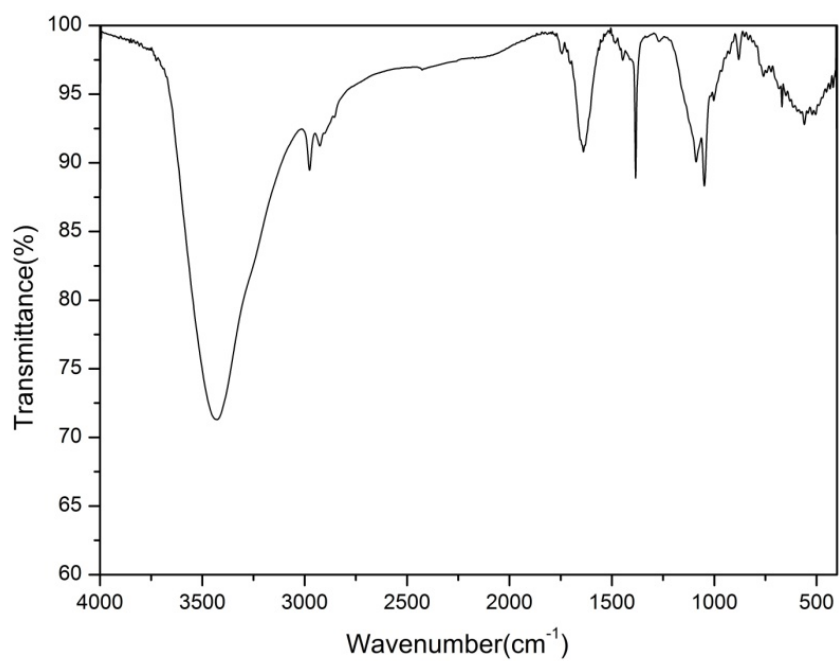


Figure S10. The IR spectrum of compound **1**.

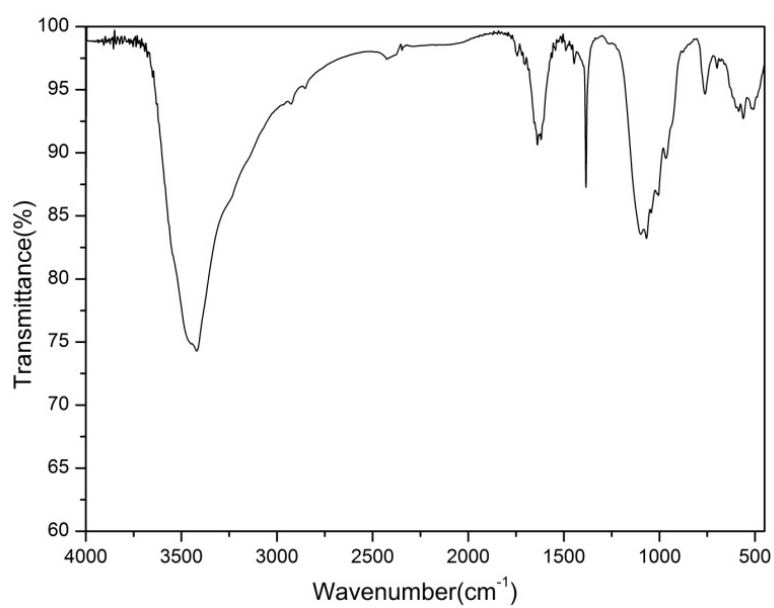


Figure S11. The IR spectrum of compound **2**.