Electronic Supplementary Information (ESI)

New series of Co, Ni, Zn, and Cd metal-organic architectures driven by an unsymmetrical biphenyl-tricarboxylic acid: Hydrothermal assembly, structural features and properties

Jin-Zhong Gu,*[a] Yan Cai,[a] Zi-Yue Qian,[a] Min Wen,[a] Zi-Fa Shi,[a] Dong-Yu Lv[a] and Alexander M. Kirillov*[b]

[a]State Key Laboratory of Applied Organic Chemistry, Key Laboratory of Nonferrous Metal Chemistry and Resources Utilization of Gansu Province, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China
Fax: (+86) 931-891-5196; E-mail: gujzh@lzu.edu.cn

[b]Centro de Química Estrutural, Complexo I, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001, Lisbon, Portugal; E-mail: kirillov@tecnico.ulisboa.pt

Synthesis and analytical data for 2–13

Synthesis of $[Zn_2(\mu\text{-Hnbtc})_2(phen)_2]\cdot 2H_2O$ (2). A mixture of $ZnCl_2$ (40.9 mg, 0.30 mmol), H_3nbtc (99.3 mg, 0.30 mmol), phen (60.0 mg, 0.3 mmol), NaOH (24 mg, 0.60 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Colourless block-shaped crystals of **2** were isolated manually, washed with distilled water and dried (yield 60% based on H_3nbtc). Anal. Calcd for $C_{54}H_{34}Zn_2N_6O_{18}$: C, 54.70; H, 2.89; N, 7.09. Found: C, 54.98; H, 2.91; N, 7.13%. IR (KBr, cm^{-1}): 3581 w, 3059 w, 1701 m, 1615 s, 1585 m, 1519 m, 1427 m, 1367 s, 1260 w, 1220 w, 1164 w, 1144 w, 1103 w, 1068 w, 915 w, 845 w, 794 w, 774 w, 758 w, 718 m, 688 w, 647 w, 576 w.

Synthesis of $[Zn(Hnbtc)(phen)_2(H_2O)]\cdot 4.5H_2O$ (3). The preparation of **3** was similar to that of **2** except using a different amount of phen (120.0 mg, 0.60 mmol). After cooling the reaction mixture to room temperature, colourless block-shaped crystals of **3** were isolated manually, washed with distilled water and dried (yield 60% based on H_3nbtc). Anal. Calcd for $C_{39}H_{34}ZnN_5O_{13.5}$: C, 54.84; H, 4.01; N, 8.20. Found: C, 54.63; H, 4.04; N, 8.17%. IR (KBr, cm^{-1}): 3561 w, 3237 w, 1702 w, 1610 s, 1580 s, 1517 m, 1424 m, 1373 s, 1291 w, 1222 w, 1141 w, 1100 w, 1066 w, 1002 w, 886 w, 846 m, 787 w, 724 m, 636 w.

Synthesis of $[Ni(Hnbtc)(phen)_2(H_2O)]\cdot 6H_2O$ (4). Synthesis of **4** was similar to **3** except using $NiCl_2\cdot 6H_2O$ instead of $ZnCl_2$. Colorless block-shaped crystals of **4** were isolated manually, washed with distilled water and dried (yield 65% based on H_3nbtc). Anal. Calcd for $C_{39}H_{37}NiN_5O_{15}$: C, 53.57; H, 4.26; N, 8.01. Found: C, 53.75; H, 4.23; N, 8.05%. IR (KBr, cm^{-1}): 3347 m, 1703 w, 1587 s, 1517 m, 1424 m, 1378 s, 1286 w, 1251 w, 1141 w, 1106 w, 1072 w, 973 w, 886 w, 846 m, 771 w, 730 m, 695 w, 643 w, 591 w.

Synthesis of $[Zn_2(\mu\text{-Hnbtc})_2(2,2'\text{-bipy})_2]\cdot 2H_2O$ (5). Synthesis of **5** was similar to **2** except using 2,2′-bipy instead of phen. Colourless block-shaped crystals of **5** were isolated manually, washed with distilled water and dried (yield 55% based on H_3nbtc). Anal. Calcd for $C_{50}H_{34}Zn_2N_6O_{18}$: C, 52.79; H,

3.01; N, 7.39. Found: C, 52.63; H, 2.99; N, 7.45%. IR (KBr, cm^{-1}): 3418 w, 3068 w, 1691 m, 1595 s, 1529 s, 1504 w, 1472 w, 1443 w, 1407 s, 1377 w, 1346 w, 1316 w, 1270 w, 1250 w, 1230 w, 1164 w, 1098 w, 1072 w, 1022 w, 936 w, 885 w, 814 w, 769 m, 733 w, 693 w, 632 w.

Synthesis of $[Cd_3(\mu_5\text{-nbtc})(\mu_6\text{-nbtc})(2,2'\text{-bipy})_2(H_2O)]_n$ (6). A mixture of $CdCl_2 \cdot H_2O$ (60.3 mg, 0.30 mmol), H_3nbtc (66.2 mg, 0.20 mmol), 2,2′-bpy (46.8 mg, 0.30 mmol), NaOH (24.0 mg, 0.60 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Colourless block-shaped crystals of **6** were isolated manually, washed with distilled water and dried (yield 62% based on H_3nbtc). Anal. Calcd for $C_{50}H_{30}Cd_3N_6O_{17}$: C, 45.35; H, 2.28; N, 6.35. Found: C, 45.31; H, 2.25; N, 6.39%. IR (KBr, cm^{-1}): 3621 w, 3074 w, 1574 s, 1524 s, 1468 m, 1407 s, 1382 s, 1336 s, 1245 w, 1164 w, 1128 w, 1093 w, 1068 w, 1017 w, 972 w, 926 w, 885 w, 845 m, 789 m, 764 m, 728 m, 698 w, 647 w.

Synthesis of $\{[Zn_3(\mu_3\text{-nbtc})_2(phen)_3(H_2O)_2]\cdot 4H_2O\}$ (7). A mixture of $ZnCl_2$ (40.9 mg, 0.30 mmol), H_3nbtc (66.2 mg, 0.20 mmol), phen (60.0 mg, 0.30 mmol), NaOH (24.0 mg, 0.60 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Colourless block-shaped crystals of **7** were isolated manually, washed with distilled water and dried (yield 60% based on H_3nbtc). Anal. Calcd for $C_{66}H_{46}Zn_3N_8O_{22}$: C, 52.87; H, 3.09; N, 7.47. Found: C, 52.73; H, 3.07; N, 7.52%. IR (KBr, cm^{-1}): 3365 m, 3063 w, 1604 s, 1586 s, 1529 s, 1517 m, 1477 w, 1424 m, 1361 s, 1222 w, 1141 w, 1106 w, 1071 w, 1048 w, 886 w, 846 m, 787 w, 724 m, 627 w, 636 w.

Synthesis of $[Co(H_2O)_6][Co_2(nbtc)_2(\mu\text{-}4,4'\text{-bipy})(4,4'\text{-bipy})_2(H_2O)_6]\cdot 8H_2O$ (8). A mixture of $CoCl_2 \cdot 6H_2O$ (71.0 mg, 0.30 mmol), H_3nbtc (66.2 mg, 0.20 mmol), 4,4′-bipy (46.8 mg, 0.30 mmol), NaOH (24.0 mg, 0.60 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Orange block-shaped crystals of **8** were isolated manually, washed with distilled water and dried (yield 56% based on H_3nbtc). Anal. Calcd for $C_{60}H_{76}Co_3N_8O_{36}$: C,

43.36; H, 4.61; N, 6.74. Found: C, 43.55; H, 4.58; N, 6.77%. IR (KBr, cm^{-1}): 3277 m, 3063 w, 1604 s, 1535 s, 1412 m, 1373 s, 1216 w, 1170 w, 1141 w, 1066 w, 1042 w, 1002 w, 811 m, 787 w, 736 w, 707 w, 666 w, 626 w.

Synthesis of $\{[Ni_3(\mu_4\text{-nbtc})_2(\mu\text{-}4,4'\text{-bipy})_{2.5}(H_2O)_4]\cdot 4H_2O\}_n$ (9). Synthesis of **9** was similar to **8** except using $NiCl_2\cdot 6H_2O$ (71.3 mg, 0.30 mmol) instead of $CoCl_2\cdot 6H_2O$. Green block-shaped crystals of **9** were isolated manually, washed with distilled water and dried (yield 63% based on H_3nbtc). Anal. Calcd for $C_{55}H_{48}Ni_3N_7O_{24}$: C, 48.32; H, 3.54; N, 7.17. Found: C, 48.11; H, 3.56; N, 7.15%. IR (KBr, cm^{-1}): 3652 w, 3292 w, 1610 s, 1533 s, 1493 w, 1412 w, 1377 s, 1220 w, 1169 w, 1133 w, 1068 w, 1012 w, 915 w, 880 w, 820 m, 784 w, 733 w, 703 w, 662 w, 637 w.

Synthesis of $\{[Cd_2(\mu_4\text{-nbtc})(\mu\text{-OH})(2,2'\text{-bipy})_2]\cdot H_2O\}_n$ (10). Synthesis of **10** was similar to **6** except using a different amount of NaOH (28.0 mg, 0.70 mmol). Colourless block-shaped crystals of **10** were obtained (yield 35% based on H_3nbtc). Anal. Calcd for $C_{35}H_{25}Cd_2N_5O_{10}$: C, 46.69; H, 2.80; N, 7.78. Found: C, 46.85; H, 2.81; N, 7.83%. IR (KBr, cm^{-1}): 3621 w, 3276 w, 1584 s, 1533 m, 1473 w, 1438 m, 1377 s, 1311 w, 1245 w, 1169 w, 1098 w, 1057 w, 1012 w, 885 w, 840 w, 764 m, 733 w, 698 w, 647 w.

Synthesis of $[Cd_2(\mu_4\text{-nbtc})(\mu\text{-OH})(phen)_2(H_2O)]_n$ (11). A mixture of $CdCl_2\cdot 4H_2O$ (60.3 mg, 0.30 mmol), H_3nbtc (66.2 mg, 0.20 mmol), phen (60.0 mg, 0.3 mmol), NaOH (28.0 mg, 0.70 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Colourless block-shaped crystals of **11** were isolated manually, washed with distilled water and dried (yield 35% based on H_3nbtc). Anal. Calcd for $C_{39}H_{25}Cd_2N_5O_{10}$: C, 49.39; H, 2.66; N, 7.38. Found: C, 49.56; H, 7.35; N, 7.45%. IR (KBr, cm^{-1}): 3660 w, 3063 w, 1575 s, 1535 m, 1511 w, 1424 m, 1373 s, 1222 w, 1170 w, 1141 w, 1095 w, 990 w, 915 w, 846 m, 782 w, 718 m, 696 w, 637 w.

Synthesis of $\{[Zn_2(\mu_5\text{-nbtc})(\mu_3\text{-OH})(\mu\text{-}4,4'\text{-bipy})]\cdot 4,4'\text{-bipy}\cdot H_2O\}_n$ (12). A mixture of $ZnCl_2\cdot H_2O$ (40.9 mg, 0.30 mmol), H_3nbtc (66.2 mg, 0.20 mmol), 4,4′-bpy (46.8 mg, 0.30 mmol), NaOH (28.0 mg, 0.70 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, then sealed in a 25 mL

Teflon-lined stainless steel vessel, and heated at 160 °C for 3 days, followed by cooling to room temperature at a rate of 10 °C·h^{-1}. Colourless block-shaped crystals of **12** were isolated manually, washed with distilled water and dried (yield 32% based on H_3nbtc). Anal. Calcd for $C_{35}H_{25}Zn_2N_5O_{10}$: C, 52.13; H, 3.12; N, 8.68. Found: C, 52.37; H, 3.09; N, 8.71%. IR (KBr, cm^{-1}): 3682 m, 3043 m, 1615 s, 1580 w, 1533 s, 1433 w, 1377 m, 1215 w, 1138 w, 1068 w, 1012 w, 1068 w, 920 w, 840 w, 804 w, 744 w, 703 w, 668 w, 642 w, 607 w.

Table S1. Selected bond lengths [Å] and angles [°] for the compounds **1**–**12**[a].

1					
Cd(1)-O(1)	2.2801(1)	Cd(1)-O(2)	2.7176(2)	Cd(1)-O(6)#1	2.3316(1)
Cd(1)-O(9)	2.3100(1)	Cd(1)-O(10)	2.3425(1)	Cd(1)-O(11)	2.3236(1)
Cd(1)-O(12)	2.2827(1)				
O(1)-Cd(1)-O(2)	51.46(1)	O(1)-Cd(1)-O(9)	91.69(1)	O(1)-Cd(1)-O(10)	81.20(1)
O(1)-Cd(1)-O(11)	110.54(1)	O(1)-Cd(1)-O(12)	96.53(1)	O(1)-Cd(1)-O(6)#1	168.36(1)
O(2)-Cd(1)-O(9)	112.36(1)	O(2)-Cd(1)-O(10)	125.85(1)	O(2)-Cd(1)-O(11)	67.52(1)
O(2)-Cd(1)-O(12)	81.17(1)	O(2)-Cd(1)-O(6)#1	140.17(1)	O(9)-Cd(1)-O(10)	90.90(1)
O(9)-Cd(1)-O(11)	82.60(1)	O(9)-Cd(1)-O(12)	166.44(1)	O(6)#1-Cd(1)-O(9)	82.50(1)
O(10)-Cd(1)-O(11)	166.63(1)	O(10)-Cd(1)-O(12)	79.78(1)	O(6)#1-Cd(1)-O(10)	88.78(1)
O(11)-Cd(1)-O(12)	104.41(1)	O(6)#1-Cd(1)-O(11)	78.83(1)	O(6)#1-Cd(1)-O(12)	87.45(1)
2					
Zn(1)-O(2)	1.9294(2)	Zn(1)-O(5)#1	1.9194(2)	Zn(1)-N(2)	2.0759(2)
Zn(1)-N(3)	2.0388(2)				
O(2)-Zn(1)-N(2)	99.19(1)	O(2)-Zn(1)-N(3)	120.90(1)	O(2)-Zn(1)-O(5)#1	111.51(1)
N(2)-Zn(1)-N(3)	81.36(1)	O(5)#1-Zn(1)-N(2)	129.62(1)	O(5)#1-Zn(1)-N(3)	112.30(1)
3					
Zn(1)-O(1)	2.0555(1)	Zn(1)-O(9)	2.1659(2)	Zn(1)-N(2)	2.1402(2)
Zn(1)-N(3)	2.1655(2)	Zn(1)-N(4)	2.1139(2)	Zn(1)-N(5)	2.1567(2)
O(1)-Zn(1)-O(9)	81.94(1)	O(1)-Zn(1)-N(2)	98.63(1)	O(1)-Zn(1)-N(3)	100.94(1)
O(1)-Zn(1)-N(4)	90.83(1)	O(1)-Zn(1)-N(5)	158.04(1)	O(9)-Zn(1)-N(2)	93.70(1)
O(9)-Zn(1)-N(3)	170.66(1)	O(9)-Zn(1)-N(4)	97.32(1)	O(9)-Zn(1)-N(5)	81.64(1)
N(2)-Zn(1)-N(3)	77.12(1)	N(2)-Zn(1)-N(4)	166.38(1)	N(2)-Zn(1)-N(5)	96.89(1)
N(3)-Zn(1)-N(4)	91.54(1)	N(3)-Zn(1)-N(5)	97.60(1)	N(4)-Zn(1)-N(5)	76.93(1)
4					
Ni(1)-O(1)	2.0682(1)	Ni(1)-O(9)	2.0763(1)	Ni(1)-N(2)	2.0775(1)
Ni(1)-N(3)	2.0975(1)	Ni(1)-N(4)	2.0645(1)	Ni(1)-N(5)	2.0802(1)
O(1)-Ni(1)-O(9)	84.09(1)	O(1)-Ni(1)-N(2)	97.35(1)	O(1)-Ni(1)-N(3)	97.35(1)
O(1)-Ni(1)-N(4)	89.46(1)	O(1)-Ni(1)-N(5)	165.92(1)	O(9)-Ni(1)-N(2)	94.71(1)
O(9)-Ni(1)-N(3)	174.32(1)	O(9)-Ni(1)-N(4)	92.57(1)	O(9)-Ni(1)-N(5)	86.84(1)
N(2)-Ni(1)-N(3)	79.67(1)	N(2)-Ni(1)-N(4)	170.51(1)	N(2)-Ni(1)-N(5)	94.10(1)
N(3)-Ni(1)-N(4)	92.94(1)	N(3)-Ni(1)-N(5)	92.74(1)	N(4)-Ni(1)-N(5)	80.20(1)
5					
Zn(1)-O(1)	1.9594(1)	Zn(1)-O(2)	2.6030(2)	Zn(1)-O(5)#1	2.0695(1)
Zn(1)-O(6)#1	2.2713(2)	Zn(1)-N(2)	2.0503(1)	Zn(1)-N(3)	2.1063(1)
O(1)-Zn(1)-O(2)	55.82(1)	O(1)-Zn(1)-N(2)	144.18(1)	O(1)-Zn(1)-N(3)	102.69(1)
O(1)-Zn(1)-O(5)#1	103.08(1)	O(1)-Zn(1)-O(6)#1	98.00(1)	O(2)-Zn(1)-N(2)	89.33(1)
O(2)-Zn(1)-N(3)	108.78(1)	O(2)-Zn(1)-O(5)#1	146.44(1)	O(2)-Zn(1)-O(6)#1	94.18(1)
N(2)-Zn(1)-N(3)	79.15(1)	O(5)#1-Zn(1)-N(2)	111.77(1)	O(6)#1-Zn(1)-N(2)	92.06(1)
O(5)#1-Zn(1)-N(3)	100.90(1)	O(6)#1-Zn(1)-N(3)	155.14(1)	O(5)#1-Zn(1)-O(6)#1	60.63(1)
6					
Cd(1)-O(1)	2.2652(1)	Cd(1)-O(4)#1	2.2596(1)	Cd(1)-O(9)	2.2577(1)
Cd(1)-O(11)	2.3657(1)	Cd(1)-O(14)	2.3816(1)	Cd(1)-O(17)	2.2764(1)
Cd(2)-O(2)	2.2214(1)	Cd(2)-O(11)	2.2953(1)	Cd(2)-O(13)	2.3952(1)
Cd(2)-O(14)	2.4180(1)	Cd(2)-N(3)	2.3146(1)	Cd(2)-N(4)	2.2743(1)
Cd(3)-O(3)	2.2553(1)	Cd(3)-O(5)#2	2.3380(1)	Cd(3)-O(6)#2	2.4077(1)
Cd(3)-O(10)#1	2.2660(1)	Cd(3)-N(5)	2.3429(1)	Cd(3)-N(6)	2.3409(1)
O(1)-Cd(1)-O(9)	95.28(1)	O(1)-Cd(1)-O(11)	91.87(1)	O(1)-Cd(1)-O(14)	82.89(1)

O(1)-Cd(1)-O(17)	168.07(1)	O(1)-Cd(1)-O(4)#1	87.86(1)	O(9)-Cd(1)-O(11)	92.74(1)
O(9)-Cd(1)-O(14)	173.35(1)	O(9)-Cd(1)-O(17)	96.58(1)	O(4)#1-Cd(1)-O(9)	95.06(1)
O(11)-Cd(1)-O(14)	80.95(1)	O(11)-Cd(1)-O(17)	88.88(1)	O(4)#1-Cd(1)-O(11)	172.18(1)
O(14)-Cd(1)-O(17)	85.47(1)	O(4)#1-Cd(1)-O(14)	91.26(1)	O(4)#1-Cd(1)-O(17)	89.77(1)
O(2)-Cd(2)-O(11)	91.65(1)	O(2)-Cd(2)-O(13)	88.38(1)	O(2)-Cd(2)-O(14)	90.78(1)
O(2)-Cd(2)-N(3)	158.93(1)	O(2)-Cd(2)-N(4)	88.03(1)	O(11)-Cd(2)-O(13)	135.85(1)
O(11)-Cd(2)-O(14)	81.62(1)	O(11)-Cd(2)-N(3)	92.19(1)	O(11)-Cd(2)-N(4)	119.47(1)
O(13)-Cd(2)-O(14)	54.25(1)	O(13)-Cd(2)-N(3)	103.06(1)	O(13)-Cd(2)-N(4)	104.66(1)
O(14)-Cd(2)-N(3)	110.28(1)	O(14)-Cd(2)-N(4)	158.90(1)	N(3)-Cd(2)-N(4)	72.09(1)
O(3)-Cd(3)-N(5)	92.81(1)	O(3)-Cd(3)-N(6)	92.89(1)	O(3)-Cd(3)-O(5)#2	137.57(1)
O(3)-Cd(3)-O(6)#2	82.25(1)	O(3)-Cd(3)-O(10)#1	104.01(1)	N(5)-Cd(3)-N(6)	70.11(1)
O(5)#2-Cd(3)-N(5)	94.98(1)	O(6)#2-Cd(3)-N(5)	92.69(1)	O(10)#1-Cd(3)-N(5)	150.09(1)
O(5)#2-Cd(3)-N(6)	128.88(1)	O(6)#2-Cd(3)-N(6)	161.96(1)	O(10)#1-Cd(3)-N(6)	84.30(1)
O(5)#2-Cd(3)-O(6)#2	55.80(1)	O(5)#2-Cd(3)-O(10)#1	89.11(1)	O(6)#2-Cd(3)-O(10)#1	113.73(1)
7					
Zn(1)-O(1)	1.9779(1)	Zn(1)-O(5)#1	1.9731(1)	Zn(1)-O(6)#1	2.6106(1)
Zn(1)-O(9)	2.0887(1)	Zn(1)-N(2)	2.0998(1)	Zn(1)-N(3)	2.1367(1)
Zn(2)-O(3)	2.0921(1)	Zn(2)-O(3)#2	2.0921(1)	Zn(2)-O(4)	2.2305(1)
Zn(2)-O(4)#2	2.2305(1)	Zn(2)-N(4)	2.0986(1)	Zn(2)-N(4)#2	2.0986(1)
O(1)-Zn(1)-O(9)	93.94(1)	O(1)-Zn(1)-N(2)	112.22(1)	O(1)-Zn(1)-N(3)	96.40(1)
O(1)-Zn(1)-O(5)#1	102.94(1)	O(1)-Zn(1)-O(6)#1	154.39(1)	O(9)-Zn(1)-N(2)	89.39(1)
O(9)-Zn(1)-N(3)	165.56(1)	O(5)#1-Zn(1)-O(9)	93.61(1)	O(6)#1-Zn(1)-O(9)	96.49(1)
N(2)-Zn(1)-N(3)	77.38(1)	O(5)#1-Zn(1)-N(2)	144.42(1)	O(6)#1-Zn(1)-N(2)	91.26(1)
O(5)#1-Zn(1)-N(3)	93.86(1)	O(6)#1-Zn(1)-N(3)	78.27(1)	O(5)#1-Zn(1)-O(6)#1	53.17(1)
O(3)-Zn(2)-O(4)	60.71(1)	O(3)-Zn(2)-N(4)	110.59(1)	O(3)-Zn(2)-O(3)#2	136.27(1)
O(3)-Zn(2)-O(4)#2	91.30(1)	O(3)-Zn(2)-N(4)#2	103.02(1)	O(4)-Zn(2)-N(4)	92.61(1)
O(4)#2-Zn(2)-N(4)	157.65(1)	N(4)-Zn(2)-N(4)#2	78.47(1)		
8					
Co(1)-O(2)	2.0999(1)	Co(1)-O(9)	2.0827(1)	Co(1)-O(10)	2.1154(1)
Co(1)-O(11)	2.0789(1)	Co(1)-N(2)	2.1926(1)	Co(1)-N(4)	2.1970(1)
Co(2)-O(12)	2.0106(1)	Co(2)-O(12)#1	2.0106(1)	Co(2)-O(13)	2.1710(1)
Co(2)-O(13)#1	2.1710(1)	Co(2)-O(14)	2.0908(1)	Co(2)-O(14)#1	2.0908(1)
O(2)-Co(1)-O(9)	91.97(1)	O(2)-Co(1)-O(10)	90.89(1)	O(2)-Co(1)-O(11)	177.64(1)
O(2)-Co(1)-N(2)	92.07(1)	O(2)-Co(1)-N(4)	90.01(1)	O(9)-Co(1)-O(10)	174.81(1)
O(9)-Co(1)-O(11)	86.47(1)	O(9)-Co(1)-N(2)	88.01(1)	O(9)-Co(1)-N(4)	86.52(1)
O(10)-Co(1)-O(11)	90.54(1)	O(10)-Co(1)-N(2)	96.22(1)	O(10)-Co(1)-N(4)	89.15(1)
O(11)-Co(1)-N(2)	89.65(1)	O(11)-Co(1)-N(4)	88.13(1)	N(2)-Co(1)-N(4)	174.21(1)
O(12)-Co(2)-O(13)	89.12(1)	O(12)-Co(2)-O(14)	89.81(1)	O(12)-Co(2)-O(13)#1	90.88(1)
O(12)-Co(2)-O(14)#1	90.19(1)	O(13)-Co(2)-O(14)	91.12(1)	O(13)-Co(2)-O(14)#1	88.88(1)
O(7)#2-Cd(2)-Cl(1)	132.23(14)	O(2)-Cd(2)-Cl(1)	90.43(12)	N(3)-Cd(2)-Cl(1)	91.82(14)
N(4)-Cd(2)-Cl(1)	123.16(12)	O(7)#2-Cd(2)-O(6)#2	52.08(16)	Cl(1)-Cd(2)-O(6)#2	87.11(12)
N(4)-Cd(2)-O(6)#2	137.63(18)	N(3)-Cd(2)-O(6)#2	80.51(18)	O(2)-Cd(2)-O(6)#2	122.51(16)
Cd(2)-Cl(1)-Cd(1)	90.15(5)				
9					
Ni(1)-O(2)	2.2273(2)	Ni(1)-O(4)#1	2.0615(2)	Ni(1)-O(12)	2.0190(2)
Ni(1)-O(18)	2.0945(2)	Ni(1)-N(5)	2.1107(2)	Ni(1)-N(7)	2.1075(2)
Ni(2)-O(2)	2.1471(2)	Ni(2)-O(9)#2	2.0559(2)	Ni(2)-O(11)	2.0442(2)
Ni(2)-O(17)	2.0797(2)	Ni(2)-O(18)	2.0869(2)	Ni(2)-N(3)	2.0757(2)
Ni(3)-O(5)	2.0578(2)	Ni(3)-O(14)#3	2.1170(2)	Ni(3)-O(19)	2.0461(2)
Ni(3)-O(20)	2.1137(2)	Ni(3)-N(6)#4	2.1063(2)	Ni(3)-N(4)#5	2.0887(2)

O(2)-Ni(1)-O(12)	84.92(1)	O(2)-Ni(1)-O(18)	77.14(1)	O(2)-Ni(1)-N(5)	100.43(1)
O(2)-Ni(1)-N(7)	167.66(1)	O(2)-Ni(1)-O(4)#1	90.74(1)	O(12)-Ni(1)-O(18)	92.77(1)
O(12)-Ni(1)-N(5)	88.93(1)	O(12)-Ni(1)-N(7)	90.43(1)	O(4)#1-Ni(1)-O(12)	174.88(1)
O(18)-Ni(1)-N(5)	176.89(1)	O(18)-Ni(1)-N(7)	91.71(1)	O(4)#1-Ni(1)-O(18)	88.90(1)
N(5)-Ni(1)-N(7)	90.89(1)	O(4)#1-Ni(1)-N(5)	89.19(1)	O(4)#1-Ni(1)-N(7)	94.36(1)
O(2)-Ni(2)-O(11)	91.15(1)	O(2)-Ni(2)-O(17)	98.98(1)	O(2)-Ni(2)-O(18)	79.11(1)
O(2)-Ni(2)-N(3)	92.96(1)	O(2)-Ni(2)-O(9)#2	170.74(1)	O(11)-Ni(2)-O(17)	87.19(1)
O(11)-Ni(2)-O(18)	88.72(1)	O(11)-Ni(2)-N(3)	173.53(1)	O(9)#2-Ni(2)-O(11)	85.77(1)
O(17)-Ni(2)-O(18)	175.46(1)	O(17)-Ni(2)-N(3)	87.23(1)	O(9)#2-Ni(2)-O(17)	89.60(1)
O(18)-Ni(2)-N(3)	96.96(1)	O(9)#2-Ni(2)-O(18)	92.08(1)	O(9)#2-Ni(2)-N(3)	90.92(1)
O(5)-Ni(3)-O(19)	93.51(1)	O(5)-Ni(3)-O(20)	93.10(1)	O(5)-Ni(3)-O(14)#3	176.83(1)
O(5)-Ni(3)-N(6)#4	91.07(1)	O(5)-Ni(3)-N(4)#5	87.73(1)	O(19)-Ni(3)-O(20)	85.36(1)
O(14)#3-Ni(3)-O(19)	89.05(1)	O(19)-Ni(3)-N(6)#4	170.95(1)	O(19)-Ni(3)-N(4)#5	93.94(1)
O(14)#3-Ni(3)-O(20)	88.99(1)	O(20)-Ni(3)-N(6)#4	86.61(1)	O(20)-Ni(3)-N(4)#5	178.95(1)
O(14)#3-Ni(3)-N(6)#4	86.66(1)	O(14)#3-Ni(3)-N(4)#5	90.21(1)	N(4)#5-Ni(3)-N(6)#4	94.03(1)
Ni(1)-O(2)-Ni(2)	94.98(1)				

10

Cd(1)-O(1)	2.2427(2)	Cd(1)-O(5)#1	2.4129(2)	Cd(1)-O(6)#1	2.4568(2)
Cd(1)-O(9)	2.3310(2)	Cd(1)-N(2)	2.3749(2)	Cd(1)-N(3)	2.3470(2)
Cd(2)-O(2)	2.3539(2)	Cd(2)-O(3)#2	2.2265(2)	Cd(2)-O(9)	2.2732(2)
Cd(2)-N(4)	2.3481(2)	Cd(2)-N(5)	2.3290(2)		
O(1)-Cd(1)-O(9)	109.42(1)	O(1)-Cd(1)-N(2)	99.19(1)	O(1)-Cd(1)-N(3)	87.80(1)
O(1)-Cd(1)-O(5)#1	91.09(1)	O(1)-Cd(1)-O(6)#1	144.59(1)	O(9)-Cd(1)-N(2)	146.39(1)
O(9)-Cd(1)-N(3)	94.06(1)	O(5)#1-Cd(1)-O(9)	101.72(1)	O(6)#1-Cd(1)-O(9)	82.26(1)
N(2)-Cd(1)-N(3)	69.24(1)	O(5)#1-Cd(1)-N(2)	94.84(1)	O(6)#1-Cd(1)-N(2)	84.53(1)
O(5)#1-Cd(1)-N(3)	163.61(1)	O(6)#1-Cd(1)-N(3)	125.59(1)	O(5)#1-Cd(1)-O(6)#1	53.52(1)
O(2)-Cd(2)-O(9)	91.21(1)	O(2)-Cd(2)-N(4)	123.14(1)	O(2)-Cd(2)-N(5)	78.41(1)
O(2)-Cd(2)-O(3)#2	84.40(1)	O(9)-Cd(2)-N(4)	140.94(1)	O(9)-Cd(2)-N(5)	103.69(1)
O(3)#2-Cd(2)-O(9)	115.20(1)	N(4)-Cd(2)-N(5)	69.99(1)	O(3)#2-Cd(2)-N(4)	88.50(1)
O(3)#2-Cd(2)-N(5)	137.64(1)	Cd(1)-O(9)-Cd(2)	111.75(1)		

11

Cd(1)-O(1)	2.3730(2)	Cd(1)-O(4)#1	2.2258(1)	Cd(1)-O(9)	2.5291(2)
Cd(1)-O(10)	2.1317(1)	Cd(1)-N(2)	2.3554(2)	Cd(1)-N(3)	2.3568(2)
Cd(2)-O(2)	2.2334(1)	Cd(2)-O(5)#2	2.5849(2)	Cd(2)-O(6)#2	2.3702(2)
Cd(2)-O(10)	2.1477(1)	Cd(2)-N(4)	2.3704(2)	Cd(2)-N(5)	2.3715(2)
O(1)-Cd(1)-O(9)	164.59(1)	O(1)-Cd(1)-O(10)	86.30(1)	O(1)-Cd(1)-N(2)	81.38(1)
O(1)-Cd(1)-N(3)	110.60(1)	O(1)-Cd(1)-O(4)#1	86.91(1)	O(9)-Cd(1)-O(10)	81.83(1)
O(9)-Cd(1)-N(2)	109.70(1)	O(9)-Cd(1)-N(3)	83.73(1)	O(4)#1-Cd(1)-O(9)	87.98(1)
O(10)-Cd(1)-N(2)	96.81(1)	O(10)-Cd(1)-N(3)	156.19(1)	O(4)#1-Cd(1)-O(10)	110.22(1)
N(2)-Cd(1)-N(3)	70.49(1)	O(4)#1-Cd(1)-N(2)	149.75(1)	O(4)#1-Cd(1)-N(3)	88.03(1)
O(2)-Cd(2)-O(10)	103.92(1)	O(2)-Cd(2)-N(4)	92.16(1)	O(2)-Cd(2)-N(5)	100.82(1)
O(2)-Cd(2)-O(5)#2	147.24(1)	O(2)-Cd(2)-O(6)#2	94.48(1)	O(10)-Cd(2)-N(4)	97.51(1)
O(10)-Cd(2)-N(5)	152.80(1)	O(5)#2-Cd(2)-O(10)	81.89(1)	O(6)#2-Cd(2)-O(10)	99.32(1)
N(4)-Cd(2)-N(5)	70.27(1)	O(5)#2-Cd(2)-N(4)	119.39(1)	O(6)#2-Cd(2)-N(4)	159.84(1)
O(5)#2-Cd(2)-N(5)	83.44(1)	O(6)#2-Cd(2)-N(5)	89.78(1)	O(5)#2-Cd(2)-O(6)#2	52.86(1)
Cd(1)-O(10)-Cd(2)					

12

Zn(1)-O(1)	2.0805(2)	Zn(1)-O(4)#1	2.1165(2)	Zn(1)-O(6)#2	2.3933(2)
Zn(1)-O(9)	2.1267(2)	Zn(1)-O(9)#3	2.0528(2)	Zn(1)-N(2)	2.0899(2)
Zn(2)-O(3)	1.9550(2)	Zn(2)-O(5)#4	1.9399(2)	Zn(2)-O(9)#5	1.9308(2)

Zn(2)-N(3)#6	2.0318(2)				
O(1)-Zn(1)-O(9)	90.65(1)	O(1)-Zn(1)-N(2)	93.76(1)	O(1)-Zn(1)-O(6)#2	173.36(1)
O(1)-Zn(1)-O(9)#3	100.31(1)	O(1)-Zn(1)-O(4)#1	85.65(1)	O(9)-Zn(1)-N(2)	99.71(1)
O(6)#2-Zn(1)-O(9)	94.48(1)	O(9)-Zn(1)-O(9)#3	81.01(1)	O(4)#1-Zn(1)-O(9)	172.73(1)
O(6)#2-Zn(1)-N(2)	81.24(1)	O(9)#3-Zn(1)-N(2)	165.92(1)	O(4)#1-Zn(1)-N(2)	86.80(1)
O(6)#2-Zn(1)-O(9)#3	84.68(1)	O(4)#1-Zn(1)-O(6)#2	89.69(1)	O(4)#1-Zn(1)-O(9)#3	93.48(1)
O(3)-Zn(2)-O(5)#4	108.20(1)	O(3)-Zn(2)-N(3)#6	110.32(1)	O(3)-Zn(2)-O(9)#5	109.97(1)
O(5)#4-Zn(2)-N(3)#6	106.06(1)	O(5)#4-Zn(2)-O(9)#5	116.36(1)	O(9)#5-Zn(2)-N(3)#6	105.80(1)
Zn(1)-O(9)-Zn(1)#3	98.74(1)	Zn(1)-O(9)-Zn(2)#7	135.66(1)	Zn(1)#3-O(9)-Zn(2)#7	104.52(1)

[a] Symmetry transformations used to generate equivalent atoms: #1 *x*, *y*+1, *z*–1 for **1** ; #1 –*x*+2, –*y*+1, –*z* for **2**; #1 –*x*+2, –*y*, –*z*+1 for **5**; #1 –*x*+1, –*y*, –*z*+2; #2 *x*, *y*, *z*+1 for **6**; #1 –*x*, *y*, –*z*; #2 –*x*+2, *y*, –*z*+1/2 for **7**; #1 –*x*+1, –*y*+1, –*z* for **8**; #1 –*x*+1, –*y*+2, –*z*+1; #2 –*x*+1, –*y*+1, –*z*; #3 –*x*, –*y*+1, –*z*–1; #4 –*x*, –*y*+1, –*z*; #5 –*x*, –*y*+2, –*z* for **9**; #1 –*x*, –*y*, –*z*; #2 –*x*+1, –*y*+1, –*z*+1 for **10**; #1 –*x*, –*y*, –*z*; #2 –*x*+1, –*y*+1, –*z*+1 for **11**; #1 –*x*, –*y*+1, –*z*; #2 *x*, *y*–1, *z*; #3 –*x*, *y*, –*z*+1/2; #4 –*x*, –*y*+2, –*z*; #5 *x*, –*y*+1, *z*–1/2; #6 –*x*+1, –*y*+1, –*z*; #7 *x*, –*y*+1, *z*+1/2 for **12**.

Table S2. Hydrogen bonds in crystal packing [Å, °] of **1**–**3**, **6**, **7**, and **9**–**13**.

Complexes	D-H…A	*d*(D-H)	*d*(H…A)	*d*(D…A)	∠DHA	Symmetry code
1	O(4)-H(1)···O(5)	0.78	1.83	2.6057(2)	173.0	*x*, *y*+1, *z*
	O(9)-H(1W)···O(13)	0.86	1.90	2.7100(2)	157.0	
	O(9)-H(2W)···O(6)	0.86	1.98	2.7239(2)	145.0	-*x*+1, -*y*+1, -*z*+1
	O(10)-H(3W)···O(1)	0.86	1.90	2.7023(2)	153.0	-*x*+1, -*y*+1, -*z*
	O(10)-H(4W)···O(3)	0.86	2.16	2.7937(2)	130.0	*x*, *y*, *z*-1
	O(11)-H(5W)···O(7)	0.87	2.13	2.9307(2)	153.0	-*x*, -*y*+1, -*z*+1
	O(11)-H(6W) ···O(9)	0.87	1.98	2.8413(2)	168.0	-*x*+1, -*y*+2, -*z*
	O(12)-H(7W) ···O(13)	0.88	1.95	2.7758(2)	157.0	*x*-1, *y*, *z*
	O(12)-H(8W) ···O(5)	0.88	2.06	2.7844(2)	140.0	*x*, *y*+1, *z*-1
	O(13)-H(9W) ···O(10)	0.85	2.05	2.8689(2)	163.0	-*x*+1, -*y*+1, -*z*
	O(13)-H(10W) ···O(2)	0.85	1.99	2.7989(2)	159.0	*x*+1, *y*, *z*
2	O(9)-H(1W)···O(6)	0.85	2.07	2.9208(3)	174.0	-*x*+2, -*y*, -*z*
	O(9)-H(2W)···O(3)	0.85	2.21	2.9185(3)	140.0	*x*+1, *y*, *z*
	O(4)-H(4)···O(1)	0.82	1.81	2.6315(3)	176.0	*x*-1, *y*, *z*
3	O(3)-H(1)···O(10)	0.82	1.84	2.5972(2)	153.0	*x*, -*y*+1, *z*+1/2
	O(9)-H(1W)···O(14)	0.91	1.89	2.7265(2)	153.0	
	O(9)-H(2W)···O(13)	0.90	2.12	2.7710(2)	128.0	
	O(10)-H(3W)···O(4)	0.85	2.01	2.8169(2)	159.0	-*x*+1/2, *y*-1/2, -*z*+1/2
	O(11)-H(5W)···O(5)	0.69	2.35	2.6675(2)	110.0	-*x*, -*y*+1, -*z*
	O(12)-H(7W)···O(10)	0.69	2.14	2.7761(2)	153.0	
	O(12)-H(8W)···O(5)	0.85	2.11	2.9600(2)	178.0	-*x*+1/2, -*y*+3/2, -*z*
	O(13)-H(9W)···O(2)	0.85	1.89	2.7377(2)	179.0	-*x*, *y*, -*z*+1/2
	O(13)-H(10W)···O(2)	0.85	1.89	2.7377(2)	179.0	
	O(14)-H(11W)···O(6)	0.85	1.85	2.7002(2)	180.0	-*x*, -*y*+1, -*z*
	O(14)-H(12W)···O(6)	0.85	1.85	2.7002(2)	180.0	*x*, -*y*+1, *z*+1/2
	O(15)-H(13W)···O(2)	0.85	1.77	2.5972(2)	163.0	-*x*+1/2, *y*-1/2, -*z*+1/2
4	O(4)-H(1)···O(11)	0.82	1.79	2.6058(2)	173.0	*x*+1, *y*, *z*
	O(9)-H(1W)···O(14)	0.86	1.90	2.7409(2)	166.0	*x*+1/2, -*y*+1/2, *z*+1/2
	O(9)-H(2W)···O(12)	0.86	2.27	3.0139(2)	146.0	-*x*+1/2, *y*-1/2, -*z*+1/2
	O(10)-H(3W)···O(14)	0.80	2.00	2.7832(2)	163.0	*x*+1/2, -*y*+1/2, *z*+1/2
	O(10)-H(4W)···O(2)	0.85	1.92	2.7711(2)	179.0	-*x*+1, -*y*, -*z*+1
	O(11)-H(5W)···O(13)	0.97	2.24	2.9879(2)	133.0	
	O(11)-H(6W)···O(3)	0.66	2.30	2.7748(2)	130.0	-*x*+1, -*y*, -*z*+1
	O(12)-H(7W)···O(2)	0.85	1.96	2.8098(2)	178.0	-*x*+1/2, *y*+1/2, -*z*+1/2
	O(14)-H(11W)···O(5)	0.86	1.82	2.6715(2)	176.0	
5	O(3)-H(1)···O(9)	0.82	1.79	2.5961(2)	166.0	*x*-1, *y*, *z*+1
	O(9)-H(1W)···O(2)	0.83	1.96	2.7637(2)	166.0	*x*, *y*, *z*-1
	O(9)-H(2W)···O(5)	0.85	1.85	2.6882(2)	171.0	-*x*+2, -*y*, -*z*+1
6	O(17)-H(1w)···O(3)	0.88	2.13	2.7889(1)	132.0	-*x*-1, -*y*+1, -*z*
	O(17)-H(2w)···O(12)	0.87	1.87	2.6275(1)	145.0	
7	O(9)-H(1W)···O(4)	0.85	1.87	2.7243(1)	179.0	*x*-1, *y*, *z*
	O(9)-H(2W)···O(2)	0.85	1.83	2.6819(1)	179.0	

	O(11)-H(5W)···O(10)	0.85	2.14	2.9919(1)	178.0	
	O(11)-H(6W)···O(2)	0.85	1.86	2.7106(1)	178.0	
8	O(9)-H(1W)···O(4)	0.82	1.87	2.6874(1)	178.0	*x*-1, *y*, *z*
	O(9)-H(2W)···O(4)	0.85	1.87	2.7233(1)	180.0	-*x*+2, -*y*, *z*+1
	O(10)-H(3W)···O(1)	0.82	1.90	2.6372(1)	150.0	
	O(10)-H(4W)···O(18)	0.85	1.90	2.7529(2)	180.0	*x*, *y*, *z*+1
	O(11)-H(5W)···O(16)	0.82	1.94	2.7437(2)	166.0	-*x*+1, -*y*+1, -*z*+1
	O(11)-H(6W)···O(3)	0.85	1.84	2.6860(1)	180.0	*x*-1, *y*, *z*
	O(12)-H(7W)···O(17)	0.82	1.93	2.6656(1)	149.0	
	O(12)-H(8W)···O(5)	0.85	1.80	2.6462(1)	180.0	-*x*+1, -*y*, -*z*+1
	O(13)-H(10W)···O(1)	0.86	2.00	2.8444(2)	167.0	-*x*+2, -*y*+1, -*z*+1
	O(14)-H(11W)···O(15)	0.82	1.85	2.6635(1)	171.0	
	O(14)-H(12W)···O(3)	0.83	2.04	2.8735(2)	179.0	-*x*+2, -*y*+1, -*z*+1
	O(15)-H(13W)···O(7)	0.86	2.13	2.9915(2)	178.0	*x*, *y*+1, *z*-1
	O(15)-H(14W)···O(1)	0.85	1.97	2.8179(2)	180.0	-*x*+2, -*y*+1, -*z*+1
	O(16)-H(15W)···O(13)	0.84	1.97	2.8188(2)	179.0	-*x*+1, -*y*+1, -*z*
	O(16)-H(16W)···O(5)	0.84	1.91	2.7486(2)	179.0	-*x*+1, -*y*+1, -*z*
	O(17)-H(17W)···O(3)	0.85	1.94	2.7897(2)	180.0	-*x*+2, -*y*+1, -*z*+1
	O(17)-H(18W)···O(6)	0.85	1.85	2.6982(1)	178.0	-*x*+1, -*y*, -*z*+1
	O(18)-H(19W)···N(3)	0.83	1.93	2.7697(2)	179.0	-*x*+1, -*y*, -*z*+1
	O(18)-H(20W)···O(10)	0.85	2.13	2.9794(2)	180.0	-*x*+2, -*y*+1, -*z*+1
9	O(17)-H(1W)···O(11)	0.82	2.59	2.8438(2)	100.0	
	O(18)-H(4W)···O(3)	0.85	1.65	2.4974(2)	179.0	-*x*+1, -*y*+2, -*z*+1
	O(19)-H(6W)···O(1)	0.85	1.92	2.7655(2)	180.0	-*x*+1, -*y*+2, -*z*
	O(20)-H(7W)···O(6)	0.82	1.93	2.6406(2)	145.0	
10	O(9)-H(1)···O(10)	0.78	2.44	2.9574(2)	124.0	
	O(10)-H(1W)···O(6)	0.83	2.30	2.9072(2)		*x*, *y*+1, *z*
	O(10)-H(2W)···O(4)	0.83	2.45	2.7399(2)		-*x*+1, -*y*+1, -*z*+1
11	O(9)-H(1W)···O(3)	0.85	1.83	2.6817(2)	180.0	-*x*, -*y*, -*z*
	O(9)-H(2W)···O(5)	0.85	2.03	2.8753(2)	179.0	*x*, *y*-1, *z*
12	O(9)-H(1)···O(2)	0.85	1.83	2.6791(2)	180.0	
	O(10)-H(1W)···O(2)	0.85	2.03	2.8782(2)	179.0	

Table S3. The emission maxima wavelengths (nm) for compounds **1**–**3**, **5**–**7**, **10**–**12** and H_3nbtc.[a]

Compound	**H_3nbtc**	**1**	**2**	**3**	**5**
λ_{em}	391, 461	402, 466	391, 464	398, 465	401, 465
Compound	**6**	**7**	**10**	**11**	**12**
λ_{em}	396, 466	404, 450	397, 464	383, 455	392, 462

[a] λ_{ex} = 271 nm for H_3nbtc and λ_{ex} = 277 nm for **1**–**3**, **5**–**7**, and **10**–**12**.

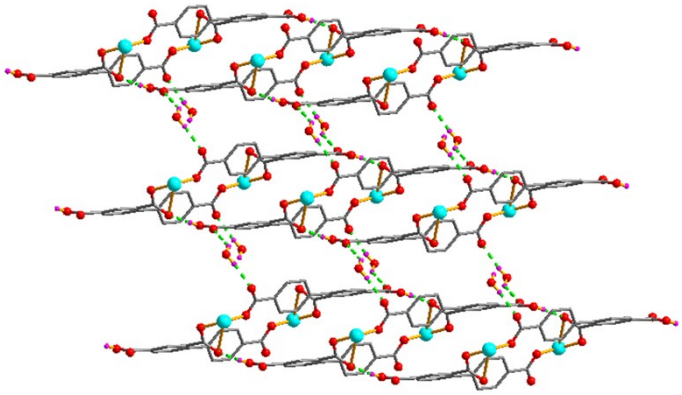


Fig. S1. 2D H-bonded network in **2** viewed along the *bc* plane (green lines present the H-bonds, NO_2 groups are omitted for clarity).

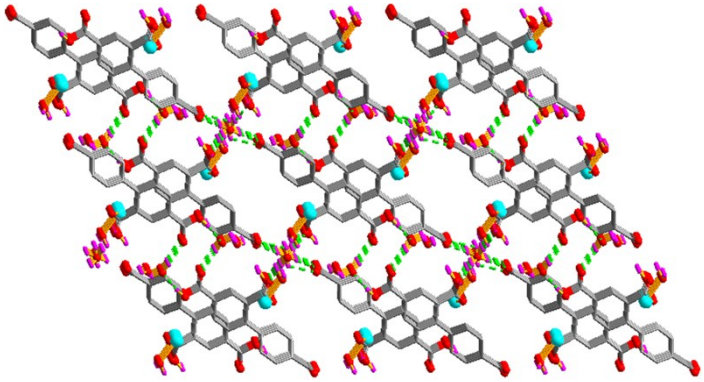


Fig. S2. 3D H-bonded network in **3** seen along the *ac* plane (green lines present the H-bonds, NO_2 groups are omitted for clarity).

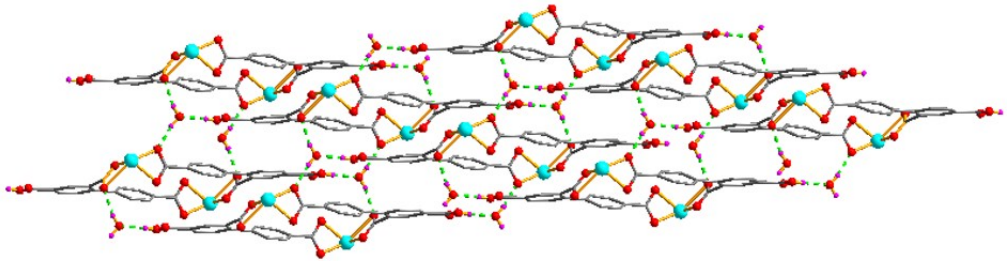
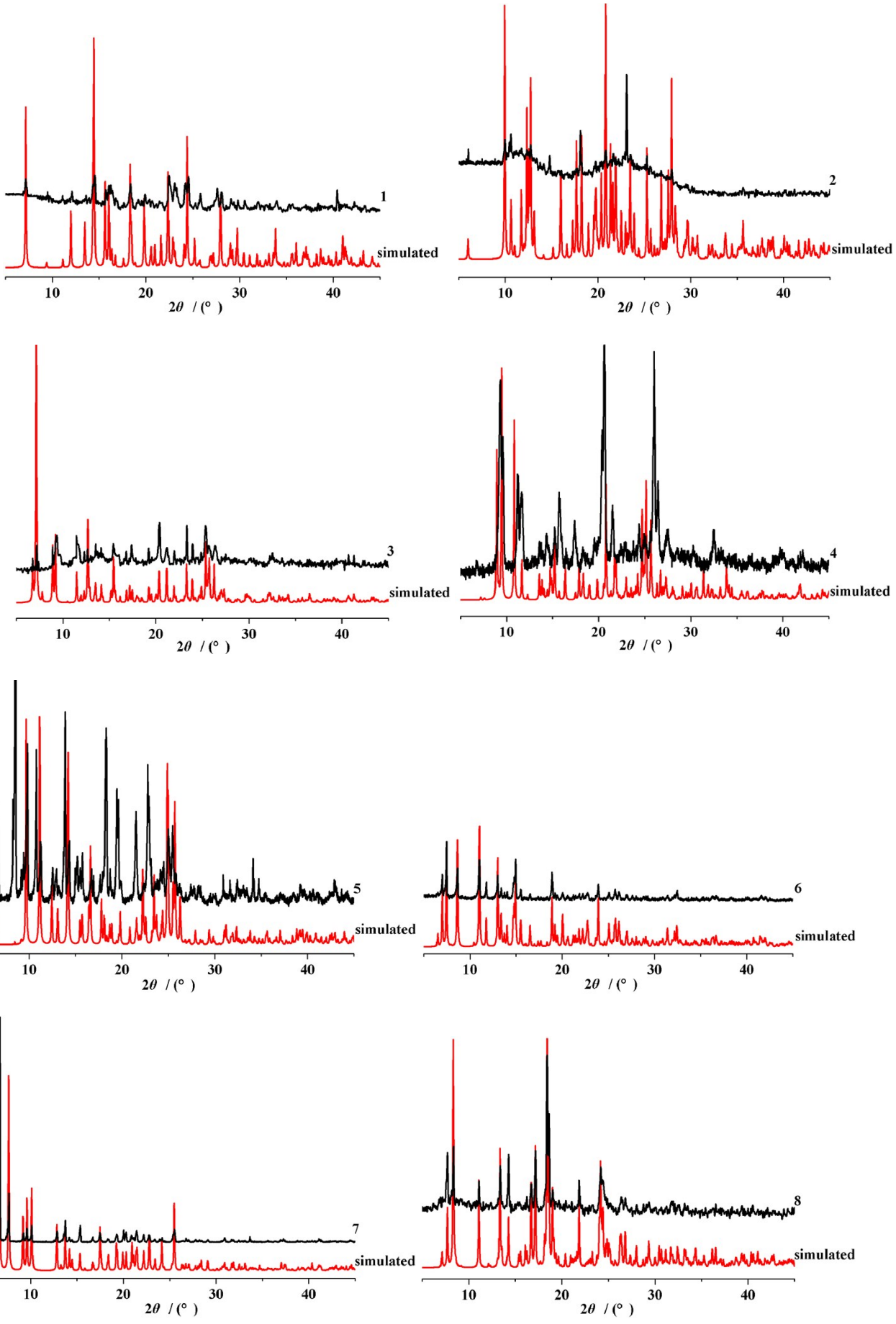


Fig. S3. 2D H-bonded network in **5** viewed along the *ac* plane (green lines present the H-bonds, NO_2 groups are omitted for clarity).


1
simulated
10
20
30
40
2θ /(°)
2
simulated
10
20
30
40
2θ /(°)
3
simulated
10
20
30
40
2θ /(°)
4
simulated
10
20
30
40
2θ /(°)
5
simulated
10
20
30
40
2θ /(°)
6
simulated
10
20
30
40
2θ /(°)
7
simulated
10
20
30
40
2θ /(°)
8
simulated
10
20
30
40
2θ /(°)

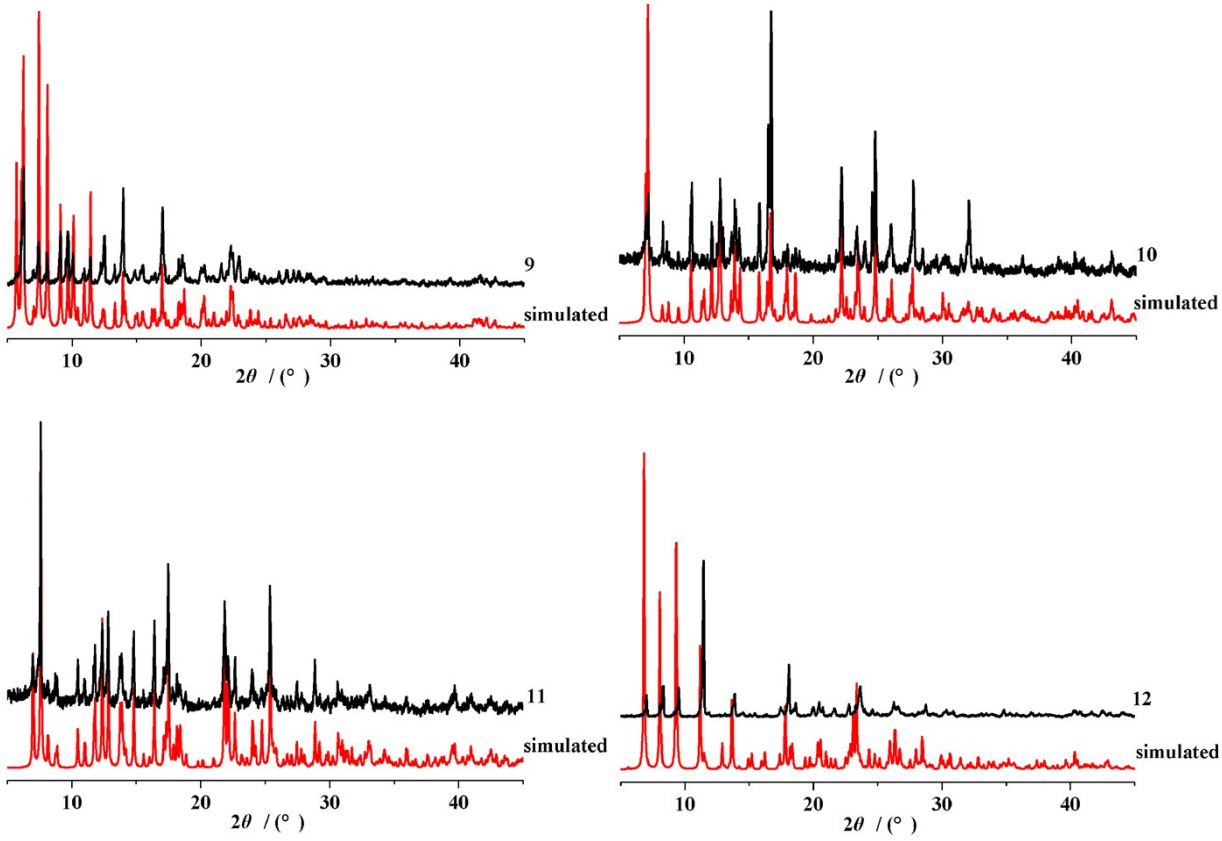


Fig. S4. PXRD patterns of compounds **1**–**12** at room temperature. Black paterns correspnond to the experimental data obtained using the as-synthesized bulk samples. Red patterns were simulated from the single crystal X-ray data.