Supporting information

Hydroisocyanurates $X_2Y(H_2C_3N_3O_3)_4{\cdot}4H_2O$ (X = K, Cs; Y = Zn, Cd) with large birefringence stemming from π−conjugated $(H_2C_3N_3O_3)^-$ anions

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Experimental methods

Synthesis. The analytical pure starting reagents KOH, $Zn(OH)_2$, $Cd(OH)_2$, Cs_2CO_3 and $H_3C_3N_3O_3$ were directly purchased from Aladdin Co., Ltd and their dissolution in deionized water took place in air.

Compounds **I** and **II** were synthesized by dissolving KOH (0.1 mmol), $Zn(OH)_2$/$Cd(OH)_2$ (0.05 mmol) and $H_3C_3N_3O_3$ (0.2 mmol) in 8 mL of deionized water in a clean hydrothermal synthesis reactor with heating using temperature-controlled furnace. The furnace was heated to 423K in 5h and left for 48h. After that, the furnace was slowly cooled at a rate of 4K/h to room temperature. Colorless crystals with a prismatic shape were obtained in the reactor. The yields of **I** and **II** are about 81.6% and 80.8% (based on KOH), respectively.

Single crystals of compound **III** were obtained by dissolving Cs_2CO_3 (0.5 mmol), $Zn(OH)_2$ (0.25 mmol) and $H_3C_3N_3O_3$ (1 mmol) in 40 mL of deionized water in a clean beaker with stirring and heating using magnetic stirrer at the boiling point until the solution was concentrated to 25 mL. Then the beaker was put in the open air to cool down naturally. Colorless crystals with a prismatic shape were obtained in the beaker.

Powder X-ray diffraction. The powder X-ray diffraction (PXRD) data of the target samples were collected on Dandong DX-2700 Focus diffractometer equipped with Cu Kα radiation ($\lambda = 1.5418$ Å) in the 2θ range of 5−70° at room temperature.

UV–vis–NIR diffuse reflectance spectrum. The UV-vis-NIR diffuse reflectance spectra were measured in the wavelength range from 200 nm to 1400 nm with polytef as reference material by Cary 7000 UV-vis-NIR universal measurement spectrophotometer under an integrating sphere. The absorption spectra were converted from reflectance spectra according to the Kubelka-Munk function: $\alpha/S = (1-R)^2/2R$, where α, S and R are the absorption, scattering and reflectance coefficients, respectively.

Infrared spectrum. Infrared (IR) spectroscopy was collected on a Varian Excalibur 3100 spectrometer in the 400−3600 cm^{-1} range. **I**, **II** and KBr samples were mixed thoroughly with mass ratio about 1:100, respectively.

Thermal analysis. Thermal gravimetric analysis (TGA) was carried out on Labsys^{TM} EVO TG-DTA/DSC thermal analyzer at a temperature range of 50−800 °C for **I** and **II** with a heating rate of 20 K/min in nitrogen atmosphere.

Single-crystal structure determination. The single-crystal X-ray diffraction data was gathered on a Rigaku AFC10 single-crystal diffractometer equipped with graphite−monochromatic Mo Kα radiation (λ = 0.71073 Å) and Saturn CCD detector at 293 K. The intensity data, data reduction and cell refinement were captured by the CrystalClear program. The crystal structures were settled by the direct method with grogram SHELXS-97 and further refined by full matrix least squares on F^2 by SHELXL-97 programs. The structure was confirmed by using the ADDSYM algorithm from the program PLATON with no higher symmetry discovered. The crystallographic data are given in Table S1–3.

Computational methods.

All theoretical calculations of $X_2Y(H_2C_3N_3O_3)_4 \cdot 4H_2O$ series (X = K, Cs; Y = Zn, Cd) were performed by *ab initio* density functional theory.[1] Notably, a similar chemical formula $K_2Mg(H_2C_3N_3O_3)_4 \cdot 4H_2O$ series have been reported.[2] Therefore, the calculating parameters for title compounds were set as same as that in early reports. The generalized gradient density approximation (GGA-PBEsol functional)[3] was adopted and the norm-conserving pseudopotentials[4] for all elements were utilized to model ion-electron interactions. K $3p^6 4s^1$, Cs $5p^6 6s^1$, Zn $4s^2 3p^6 3d^{10}$, Cd $5s^2 4p^6 4d^{10}$, C $2s^2 2p^2$, N $2s^2 2p^3$, O $2s^2 2p^4$ and H $1s^1$ electrons were treated as the valence electrons, respectively. The kinetic energy cutoff of 770 eV and Monkhorst-Pack *k*-point meshes[5] (2×2×4) in the first Brillouin zone were chosen to ensure the accuracy of the calculated results. The scissors operator, set as the difference between the experimental and PBEsol bandgaps, was adopted in birefringence calculations. The structural parameters (lattice constant and atomic position) was fixed without any optimization. In early reports, this method has been applied on H-free metal cyanurates as well as H-containing hydro-cyanurates successfully.[6-9]

Table S1. Crystal data and structure refinement for **I**, **II**, and **III**.

Empirical formula	$K_2Zn(H_2C_3N_3O_3)_4 \cdot 4H_2O$	$K_2Cd(H_2C_3N_3O_3)_4 \cdot 4H_2O$	$Cs_2Zn(H_2C_3N_3O_3)_4 \cdot 4H_2O$
Formula weight	727.94	774.97	915.56
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	*C*2/*m*	*C*2/*m*	*C*2/*m*
a/Å	11.7653 (6)	10.6404(6)	11.3093(9)
b/Å	15.9132 (8)	16.1413(10)	16.4363(10)
c/Å	6.9886 (3)	7.0699(4)	7.1029(5)
α/°	90	90	90
β/°	116.846 (5)	99.247(6)	104.568(8)
γ/°	90	90	90
Volume/$Å^3$	1167.41 (10)	1198.47(12)	1277.86(16)
Z	2	2	2
ρ_{calc}/g/cm^3	2.071	2.148	2.379
μ/mm^{-1}	1.518	1.366	3.869
F(000)	736.0	772.0	880.0
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
Index ranges	$-14 \le h \le 14$, $-19 \le k \le 19$, $-8 \le l \le 8$	$-13 \le h \le 13$, $-20 \le k \le 17$, $-8 \le l \le 8$	$-14 \le h \le 12$, $-18 \le k \le 20$, $-8 \le l \le 8$
Reflections collected	6563	6523	4778
Goodness-of-fit on F^2	1.170	1.113	1.046
Final R indexes	$R_1 = 0.0180$	$R_1 = 0.0197$	$R_1 = 0.0312$
[I>=2σ (I)]	$wR_2 = 0.0567$	$wR_2 = 0.0514$	$wR_2 = 0.0827$
Final R indexes	$R_1 = 0.0187$	$R_1 = 0.0198$	$R_1 = 0.0332$
[all data]	$wR_2 = 0.0569$	$wR_2 = 0.0515$	$wR_2 = 0.0840$

Table S2. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for **I**, **II,** and **III**.

I

Atom	**x**	**y**	**z**	**Ueq**
K1	0.14468 (4)	0.5000	0.36744 (8)	0.02648 (14)
Zn1	0.5000	0.5000	0.5000	0.01620 (13)
C1	0.60344 (12)	0.68103 (8)	0.5147 (2)	0.0145 (3)
C2	0.39950 (12)	0.31564 (9)	−0.0062 (2)	0.0156 (3)
C3	0.5000	0.81520 (12)	0.5000	0.0179 (4)
C4	0.5000	0.18206 (12)	0.0000	0.0185 (4)
O1	0.70084 (9)	0.64674 (6)	0.52930 (17)	0.0214 (2)
O2	0.40768 (14)	0.5000	0.7050 (2)	0.0213 (3)
O3	0.30478 (9)	0.35178 (6)	−0.01044 (17)	0.0226 (2)
O4	0.32386 (14)	0.5000	0.2095 (2)	0.0214 (3)
O5	0.5000	0.89196 (9)	0.5000	0.0283 (4)
O6	0.5000	0.10529 (9)	0.0000	0.0288 (4)
N1	0.5000	0.63749 (10)	0.5000	0.0148 (3)
N2	0.5000	0.35885 (10)	0.0000	0.0171 (3)
N3	0.59967 (10)	0.76810 (7)	0.51125 (19)	0.0181 (3)
N4	0.40082 (10)	0.22897 (7)	−0.00735 (19)	0.0187 (3)

II

Atom	**x**	**x**	**z**	**Ueq**
K1	0.85887 (6)	0.5000	0.71616 (9)	0.02939 (15)
Cd1	0.5000	0.5000	0.5000	0.01919 (10)
C1	0.39632 (15)	0.31643 (10)	0.4116 (2)	0.0160 (3)
C2	0.60038 (15)	0.31371 (10)	0.0942 (2)	0.0168 (3)
C3	0.5000	0.18428 (15)	0.5000	0.0192 (5)
C4	0.5000	0.18152 (15)	0.0000	0.0195 (5)
O1	0.29936 (11)	0.35182 (8)	0.33098 (19)	0.0234 (3)
O4	0.69042 (18)	0.5000	0.3683 (3)	0.0254 (4)
O2	0.60396 (19)	0.5000	0.8237 (3)	0.0257 (4)

O3	0.69550 (11)	0.34901 (8)	0.18398 (19)	0.0238 (3)
O5	0.5000	0.10876 (11)	0.5000	0.0290 (4)
O6	0.5000	0.10604 (11)	0.0000	0.0302 (4)
N1	0.5000	0.35871 (12)	0.5000	0.0164 (4)
N2	0.5000	0.35626 (12)	0.0000	0.0180 (4)
N3	0.40011 (13)	0.23074 (9)	0.4110 (2)	0.0195 (3)
N4	0.59896 (13)	0.22806 (9)	0.0923 (2)	0.0200 (3)

III

Atom	**x**	**y**	**z**	**Ueq**
Cs1	0.333993 (18)	1.0000	0.17547 (4)	0.03765 (19)
Zn1	0.5000	0.5000	0.5000	0.0242 (2)
C1	0.3995 (2)	0.67857 (13)	0.4058 (3)	0.0234 (5)
C2	0.09973 (19)	0.81094 (13)	0.0986 (3)	0.0223 (5)
C3	0.0000	0.68197 (18)	0.0000	0.0232 (7)
C4	0.5000	0.80792 (19)	0.5000	0.0270 (7)
N1	0.5000	0.63624 (14)	0.5000	0.0222 (6)
N2	0.0000	0.85248 (14)	0.0000	0.0250 (6)
N3	0.40179 (17)	0.76199 (10)	0.4060 (3)	0.0263 (5)
N4	0.09746 (16)	0.72694 (10)	0.0962 (3)	0.0256 (5)
O1	0.3119 (2)	0.5000	0.4945 (4)	0.0310 (6)
O2	0.5523 (3)	0.5000	0.8054 (4)	0.0314 (6)
O3	0.0000	0.60765 (13)	0.0000	0.0367 (6)
O4	0.30333 (15)	0.64475 (9)	0.3164 (3)	0.0326 (5)
O5	0.5000	0.88163 (13)	0.5000	0.0436 (7)
O6	0.19371 (15)	0.84486 (9)	0.1921 (3)	0.0337 (5)

Table S3. Bond lengths (Å) and angles (degree) for **I**, **II** and **III**.

I			
K1—O1$^{\mathrm{i}}$	2.8448 (10)	Zn1—N1	2.1879 (16)
K1—O1$^{\mathrm{ii}}$	2.8448 (10)	C1—O1	1.2311 (17)
K1—O2	2.9234 (16)	C1—N1	1.3633 (15)
K1—O4	2.7865 (16)	C1—N3	1.3862 (18)
K1—O5$^{\mathrm{iii}}$	2.8523 (9)	C2—O3	1.2425 (17)
K1—O5$^{\mathrm{iv}}$	2.8523 (9)	C2—N2	1.3515 (16)
K1—O6$^{\mathrm{v}}$	2.8899 (9)	C2—N4	1.3792 (19)
K1—O6$^{\mathrm{vi}}$	2.8899 (9)	C3—O5	1.222 (2)
Zn1—O2$^{\mathrm{i}}$	2.1533 (14)	C3—N3$^{\mathrm{ii}}$	1.3638 (15)
Zn1—O2	2.1533 (14)	C3—N3	1.3637 (15)
Zn1—O4	2.1486 (15)	C4—O6	1.222 (2)
Zn1—O4$^{\mathrm{i}}$	2.1486 (15)	C4—N4$^{\mathrm{vii}}$	1.3666 (16)
Zn1—N1$^{\mathrm{i}}$	2.1879 (16)	C4—N4	1.3666 (16)
O1—C1—N1	123.15 (13)	N3—C3—N3$^{\mathrm{ii}}$	113.32 (16)
O1—C1—N3	117.75 (12)	O6—C4—N4	123.11 (8)
N1—C1—N3	119.09 (12)	O6—C4—N4$^{\mathrm{vii}}$	123.11 (8)
O3—C2—N2	121.84 (13)	N4—C4—N4$^{\mathrm{vii}}$	113.78 (17)
O3—C2—N4	118.36 (12)	C1$^{\mathrm{ii}}$—N1—C1	118.92 (16)
N2—C2—N4	119.80 (12)	C2$^{\mathrm{vii}}$—N2—C2	118.84 (17)
O5—C3—N3	123.34 (8)	C3—N3—C1	124.77 (12)
O5—C3—N3$^{\mathrm{ii}}$	123.34 (8)	C4—N4—C2	123.89 (12)

Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) −x+1, y, −z+1; (iii) x−1/2, y−1/2, z; (iv) −x+1/2, −y+3/2, −z+1; (v) x−1/2, y+1/2, z; (vi) −x+1/2, −y+1/2, −z; (vii) −x+1, y, −z.

II

K1—O1^{i}	2.9131 (13)	C1—O1	1.236 (2)
K1—O1^{ii}	2.9131 (13)	C1—N1	1.3607 (18)
K1—O4	2.801 (2)	C1—N3	1.384 (2)
K1—O2	2.932 (2)	C2—O3	1.243 (2)
K1—O5^{iii}	2.9000 (12)	C2—N2	1.3518 (19)
K1—O5^{iv}	2.9000 (12)	C2—N4	1.383 (2)
K1—O6^{v}	2.8711 (12)	C3—O5	1.219 (3)
K1—O6^{iii}	2.8711 (12)	C3—N3^{i}	1.3686 (19)
Cd1—O4	2.3619 (19)	C3—N3	1.3686 (19)
Cd1—O4^{ii}	2.3619 (19)	C4—O6	1.218 (3)
Cd1—O2	2.3767 (19)	C4—N4	1.3703 (19)
Cd1—O2^{ii}	2.3767 (19)	C4—N4^{vi}	1.3703 (19)
Cd1—N1^{ii}	2.2806 (19)	N1—C1^{i}	1.3608 (18)
Cd1—N1	2.2807 (19)	N2—C2^{vi}	1.3518 (19)
O1—C1—N1	122.37 (16)	N3—C3—N3^{i}	113.6 (2)
O1—C1—N3	118.90 (15)	O6—C4—N4	123.24 (11)
N1—C1—N3	118.73 (15)	O6—C4—N4^{vi}	123.24 (11)
O3—C2—N2	122.19 (16)	N4^{vi}—C4—N4	113.5 (2)
O3—C2—N4	118.04 (15)	C1—N1—C1^{i}	119.8 (2)
N2—C2—N4	119.77 (15)	C2^{vi}—N2—C2	118.9 (2)
O5—C3—N3^{i}	123.22 (10)	C3—N3—C1	124.55 (15)
O5—C3—N3	123.22 (10)	C4—N4—C2	124.00 (15)

Symmetry codes: (i) −x+1, y, −z+1; (ii) −x+1, −y+1, −z+1; (iii) −x+3/2, −y+1/2, −z+1; (iv) x+1/2, y+1/2, z; (v) x+1/2, y+1/2, z+1; (vi) −x+1, y, −z.

III			
Cs1—O1$^{\mathrm{iv}}$	3.188 (3)	C1—N1	1.357 (2)
Cs1—O2$^{\mathrm{v}}$	3.584 (3)	C1—N3	1.371 (3)
Cs1—O3$^{\mathrm{iii}}$	3.0636 (13)	C1—O4	1.244 (3)
Cs1—O3$^{\mathrm{ii}}$	3.0636 (13)	C2—N2	1.353 (2)
Cs1—O5$^{\mathrm{i}}$	3.2321 (14)	C2—N4	1.381 (3)
Cs1—O5	3.2321 (14)	C2—O6	1.235 (2)
Cs1—O6	3.0212 (16)	C3—N4$^{\mathrm{viii}}$	1.360 (2)
Cs1—O6$^{\mathrm{vi}}$	3.0211 (16)	C3—N4	1.360 (2)
Zn1—N1$^{\mathrm{vii}}$	2.239 (2)	C3—O3	1.222 (4)
Zn1—N1	2.239 (2)	C4—N3$^{\mathrm{ix}}$	1.370 (2)
Zn1—O1	2.118 (2)	C4—N3	1.370 (2)
Zn1—O1$^{\mathrm{vii}}$	2.118 (2)	C4—O5	1.212 (4)
Zn1—O2	2.100 (3)	N1—C1$^{\mathrm{ix}}$	1.357 (2)
Zn1—O2$^{\mathrm{vii}}$	2.100 (3)	N2—C2$^{\mathrm{viii}}$	1.353 (2)
N1—C1—N3	119.92 (19)	O3—C3—N4$^{\mathrm{viii}}$	122.93 (13)
O4—C1—N1	122.6 (2)	N3—C4—N3$^{\mathrm{ix}}$	113.1 (3)
O4—C1—N3	117.46 (19)	O5—C4—N3$^{\mathrm{ix}}$	123.45 (13)
N2—C2—N4	119.18 (19)	O5—C4—N3	123.45 (13)
O6—C2—N2	122.9 (2)	C1—N1—C1$^{\mathrm{ix}}$	118.3 (2)
O6—C2—N4	117.94 (18)	C2$^{\mathrm{viii}}$—N2—C2	119.4 (2)
N4—C3—N4$^{\mathrm{viii}}$	114.1 (3)	C4—N3—C1	124.36 (19)
O3—C3—N4	122.92 (13)	C3—N4—C2	124.04 (18)

Symmetry codes: (i) −x+1, −y+2, −z+1; (ii) x+1/2, y+1/2, z; (iii) −x+1/2, −y+3/2, −z; (iv) −x+1/2, −y+3/2, −z+1; (v) x−1/2, y+1/2, z−1; (vi) x, −y+2, z; (viii) x−1/2, y−1/2, z; (viii) −x, y, −z; (ix) −x+1, y, −z+1.

Table S4. Hydrogen-bond information for **I**, **II** and **III** (Å, º)

I

D—H···A	d (D—H)	d (H···A)	d (D···A)	< (D—H···A)
N3—H3···O1$^{\mathrm{i}}$	0.86	1.98	2.8331 (14)	170
N4—H4A···O3$^{\mathrm{ii}}$	0.86	1.94	2.7910 (15)	173
O2—H2···N2$^{\mathrm{iii}}$	0.84 (2)	2.08 (2)	2.9075 (16)	169.1 (19)
O4—H4···O3	0.83 (2)	1.94 (2)	2.7684 (13)	171 (2)

II

D—H···A	d (D—H)	d (H···A)	d (D···A)	< (D—H···A)
N3—H3···O3$^{\mathrm{iv}}$	0.86	1.95	2.8018 (18)	173
N4—H4A···O1$^{\mathrm{v}}$	0.86	1.96	2.8109 (19)	170
O2—H2···N2$^{\mathrm{vi}}$	0.81 (3)	2.12 (3)	2.933 (2)	176 (3)
O4—H4···O3$^{\mathrm{vii}}$	0.80 (3)	1.99 (3)	2.7682 (16)	167 (3)

III

D—H···A	d (D—H)	d (H···A)	d (D···A)	< (D—H···A)
N3—H3···O6	0.86	1.95	2.811 (2)	178
N4—H4···O4	0.86	1.94	2.803 (2)	177
O2—H2···N2$^{\mathrm{viii}}$	0.73 (3)	2.21 (3)	2.924 (2)	167 (4)
O1—H1···O4	0.86 (4)	1.92 (4)	2.685 (2)	147 (4)

Symmetry codes: (i) −x+3/2, −y+3/2, −z+1; (ii) −x+1/2, −y+1/2, −z; (iii) x, y, z+1; (iv) x−1/2, −y+1/2, z; (v) x+1/2, −y+1/2, z; (vi) −x+1, −y+1, −z+1; (vii) x, −y+1, z; (viii) −x+1/2, −y+3/2, −z+1.

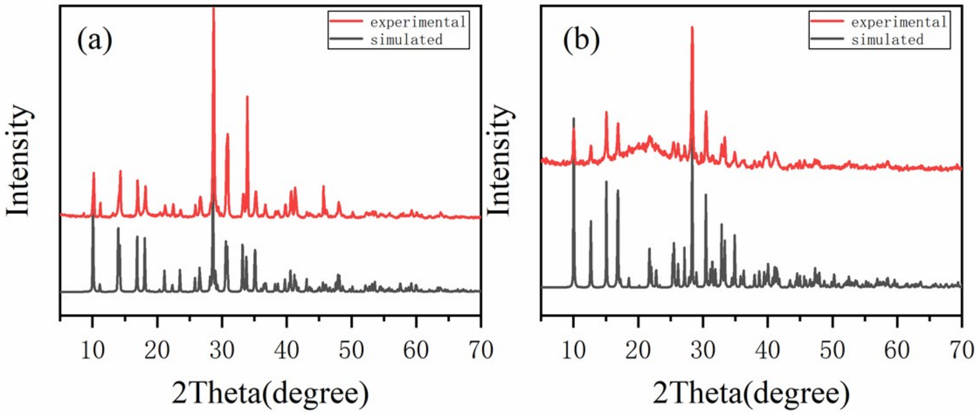

Figure S1. The calculated and experimental PXRD patterns for **I** (a) and **II** (b).

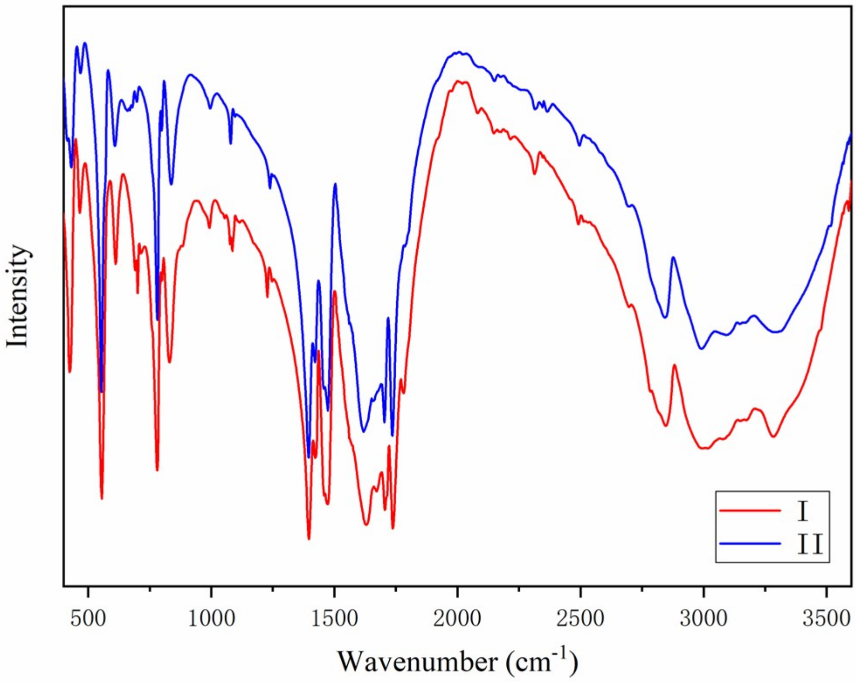

Figure S2. The IR spectra for **I** and **II**

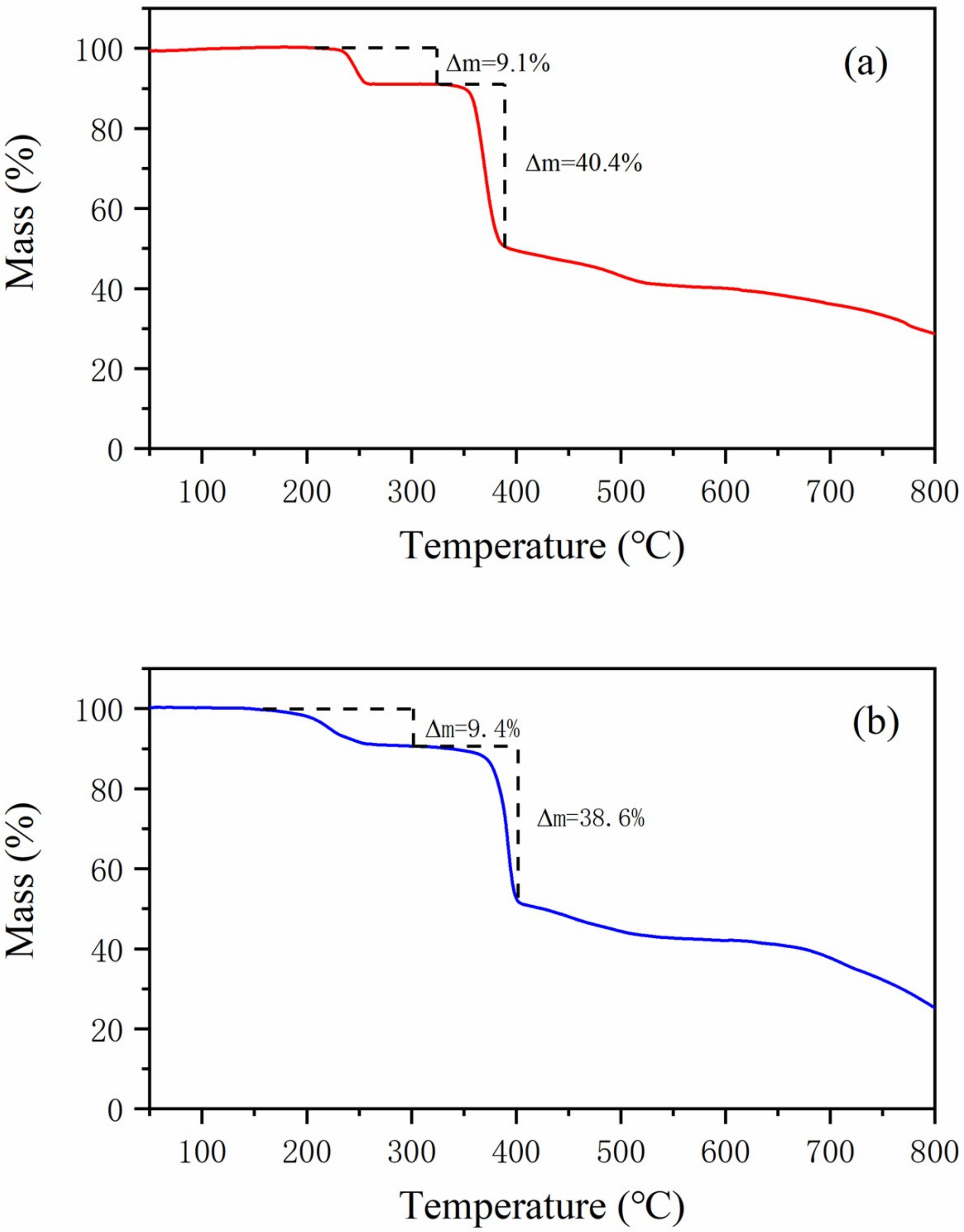

Figure S3. The TG curves for **I** (a) and **II** (b).

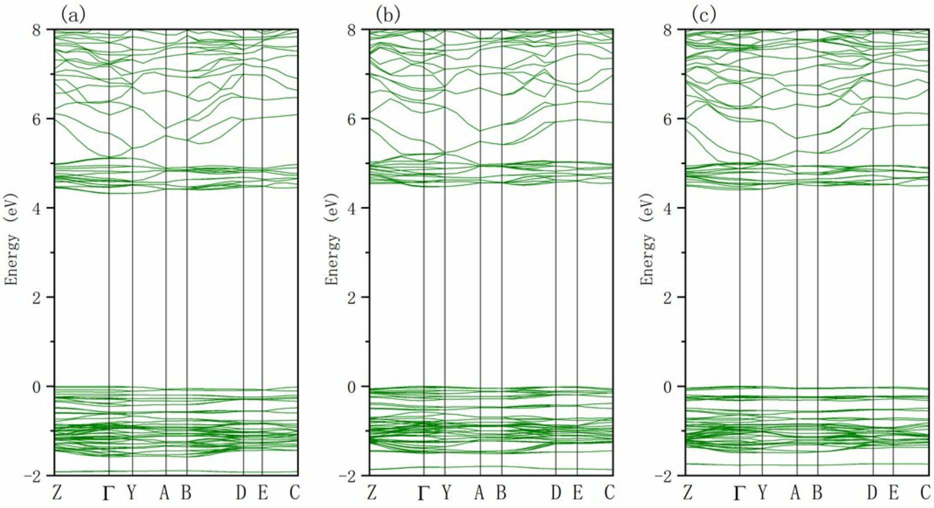

Figure S4. The simulated bandgaps of **I** (a), **II** (b), and **III** (c).

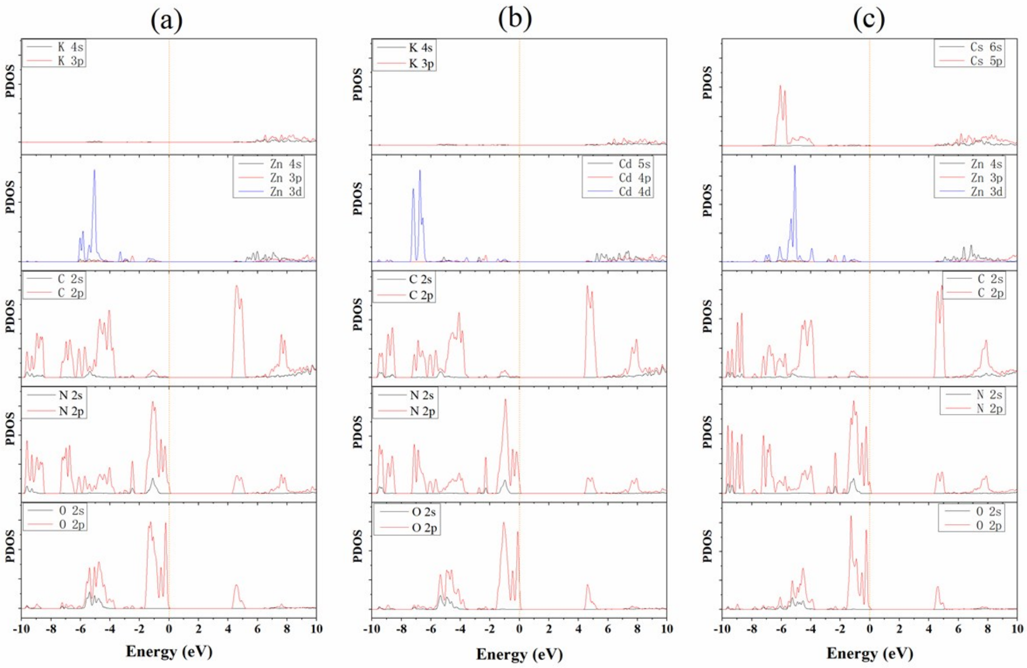

Figure S5. The simulated DOS of **I** (a), **II** (b), and **III** (c).

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