Supporting Information

Regulation of structure and density of KN_5 framework *via* CH_3COOK and CF_3COOK

Shuaijie Jiang, Jianxin Zhou, Yuangang Xu*, Qiuhan Lin, Pengcheng Wang*, Ming Lu

School of Chemistry and Chemical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

Corresponding Author

Yuangang Xu: yuangangxu@163.com

Pengcheng Wang: alexwpch@njust.edu.cn

Table of Contents

1. Single-crystal X-ray diffraction analysis

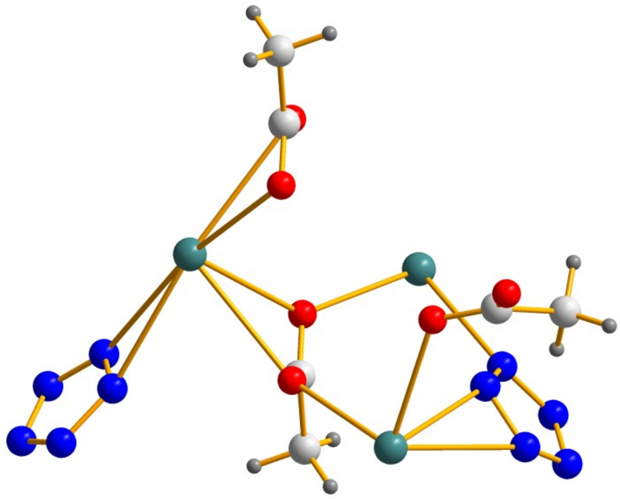

Fig. S1 The asymmetric unit of framework **1**.

Table S1 Crystallographic data of compound **1**

CCDC	2208012
Crystal data	
Chemical formula	$C_6H_9K_5N_{10}O_6$
M_r	512.73
Crystal system, space group	Trigonal, $P\bar{3}c1$
Temperature (K)	243
a, *c* (Å)	10.3592 (2), 20.7675 (8)
V (Å^3)	1930.04 (11)
Z	4
Radiation type	Mo *Kα*
m (mm^{-1})	1.19
Crystal size (mm)	0.13 × 0.12 × 0.1
Data collection	
Diffractometer	Bruker *APEX*-II CCD
Absorption correction	Multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0732 before and 0.0552 after correction. The Ratio of minimum to maximum transmission is 0.8836. The l/2 correction factor

	is Not present.
T_{min}, T_{max}	0.659, 0.745
No. of measured, independent and observed [$I > 2s(I)$] reflections	10672, 1152, 985
R_{int}	0.046
(sin q/l)$_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2s(F^2)]$, $wR(F^2)$, S	0.027, 0.062, 1.14
No. of reflections	1152
No. of parameters	143
No. of restraints	105
H-atom treatment	H-atom parameters constrained
Dρ_{max}, Dρ_{min} (e Å^{-3})	0.28, -0.32

Table S2. Bond lengths [Å] for **1**.

K001—K002i	4.5045 (12)	K003—O005	2.8627 (15)
K001—K003ii	3.8042 (6)	K003—O005iii	3.0061 (17)
K001—K003iii	3.8042 (6)	K003—N5	2.9340 (11)
K001—K003iv	3.8042 (6)	K003—N4	3.20 (4)
K001—O004v	2.6833 (15)	O004—C006	1.259 (2)
K001—O004vi	2.6833 (15)	O005—C006	1.255 (3)
K001—O004	2.6833 (15)	C006—C007	1.506 (3)
K001—N008	3.011 (12)	C007—H00A	0.9700
K001—N1	3.069 (12)	C007—H00B	0.9700
K002—K003vii	3.8038 (6)	C007—H00C	0.9700
K002—K003viii	3.8038 (6)	N009—N008	1.294 (7)
K002—K003iii	3.8038 (6)	N009—N2	1.327 (8)

K002—O005ix	2.6830 (15)	N008—N1	1.318 (7)
K002—O005	2.6830 (15)	N1—N3	1.316 (7)
K002—O005x	2.6830 (16)	N3—N2	1.298 (7)
K002—N009	2.763 (6)	N5—N4	1.302 (17)
K003—K003iii	3.6305 (10)	N5—N8	1.312 (13)
K003—O004iii	3.0098 (17)	N4—N6	1.302 (16)
K003—O004	2.8637 (15)	N6—N9	1.306 (13)
K003—O004viii	2.9554 (16)	N8—N9	1.302 (13)

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

Table S3. Torsion angles [°] of **1**.

K001—O004—C006—O005	−120.2 (2)	K003i—O005—C006—C007	−142.04 (19)
K001—O004—C006—C007	59.7 (3)	K003—O005—C006—C007	141.58 (19)
K001—N008—N1—N3	159.4 (5)	K003—N5—N4—N6	−158 (2)
K001—N1—N3—N2	93 (3)	K003—N5—N8—N9	127 (4)
K002—O005—C006—O004	120.5 (2)	K003—N4—N6—N9	−71 (6)
K002—O005—C006—C007	−59.3 (3)	N009—N008—N1—K001	−158.8 (5)
K002—N009—N008—K001	54 (3)	N009—N008—N1—N3	0.6 (7)
K002—N009—N008—N1	163.7 (5)	N008—N009—N2—N3	0.0 (7)
K002—N009—N2—N3	−164.4 (6)	N008—N1—N3—N2	−0.6 (7)
K003i—O004—C006—O005	−37.8 (2)	N1—N3—N2—N009	0.4 (7)
K003ii—O004—C006—O005	134.57 (17)	N2—N009—N008—K001	−110 (3)
K003—O004—C006—O005	38.5 (2)	N2—N009—N008—N1	−0.3 (7)
K003—O004—C006—C007	−141.60 (19)	N5—N4—N6—N9	0.0 (3)
K003ii—O004—C006—C007	−45.5 (2)	N5—N8—N9—N6	−0.1 (7)
K003i—O004—C006—C007	142.10 (19)	N4—N5—N8—N9	0.0 (6)
K003i—O005—C006—O004	37.8 (2)	N4—N6—N9—N8	0.1 (6)
K003—O005—C006—O004	−38.5 (2)	N8—N5—N4—K003	158 (2)

K003iii—O005—C006—O004	−134.61 (17)	N8—N5—N4—N6	0.0 (3)
K003iii—O005—C006—C007	45.5 (2)		

Symmetry code(s): (i) −x+1, −y+1, −z+1; (ii) x−y, x, −z+1; (iii) y, −x+y, −z+1.

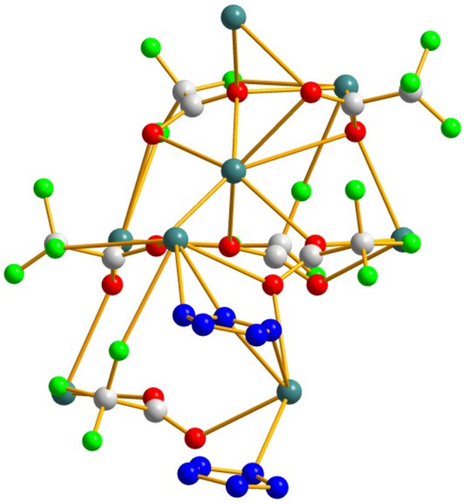

Fig. S2 The asymmetric unit of framework **2**.

Table S4 Crystallographic data of compound **2**

CCDC	2208013
Crystal data	
Chemical formula	$C_{12}F_{18}K_8N_{10}O_{12}$
M_r	1131.02
Crystal system, space group	Orthorhombic, *Pna*2$_1$
Temperature (K)	170
a, *b*, *c* (Å)	23.7161 (16), 7.1980 (7), 20.5953 (18)
V (Å^3)	3515.8 (5)
Z	4
Radiation type	Mo *K*a
m (mm^{-1})	1.14
Crystal size (mm)	0.15 × 0.08 × 0.05
Data collection	
Diffractometer	Bruker *APEX*-II CCD
Absorption correction	Multi-scan

	SADABS2016/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0903 before and 0.0693 after correction. The Ratio of minimum to maximum transmission is 0.8578. The l/2 correction factor is Not present.
T_{min}, T_{max}	0.639, 0.745
No. of measured, independent and observed [I > 2s(I)] reflections	20259, 5798, 3453
R_{int}	0.074
(sin q/l)$_{max}$ (Å^{-1})	0.626
Refinement	
$R[F^2 > 2s(F^2)]$, $wR(F^2)$, S	0.073, 0.227, 1.02
No. of reflections	5798
No. of parameters	569
No. of restraints	56
	$w = 1/[s^2(F_o^2) + (0.110P)^2 + 11.5328P]$ where $P = (F_o^2 + 2F_c^2)/3$
Dρ_{max}, Dρ_{min} (e Å^{-3})	1.26, -0.53
Absolute structure	Flack x determined using 984 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	-0.01 (4)

Table S5 Bond lengths [Å] for **2**.

K5—K6	3.616 (5)	O7—K13	2.92 (6)
K5—K6^{i}	3.582 (5)	F15—C10	1.321 (15)
K5—O3	2.834 (10)	F10—C7	1.304 (17)
K5—O9	2.784 (10)	F14—C10	1.288 (14)
K5—O2	2.789 (10)	F14—K11^{vii}	3.32 (6)
K5—O11^{ii}	2.825 (10)	F12—C7	1.297 (17)
K5—O5	2.837 (11)	F12—K10^{iv}	3.29 (6)
K5—O7	2.805 (11)	F7—C6	1.321 (15)

K5—O12^{ii}	3.005 (12)	F4—C4	1.306 (14)
K5—O4	3.071 (11)	F4—K10^{ii}	3.35 (6)
K5—O6	3.012 (11)	O8—C8	1.286 (15)
K6—O3	2.787 (10)	O8—K11	2.41 (6)
K6—O9^{ii}	2.819 (10)	F18—C11	1.337 (12)
K6—O2^{ii}	2.844 (11)	O1—C2	1.259 (13)
K6—O11^{ii}	2.767 (11)	O1—K9	2.57 (5)
K6—O5	2.771 (11)	F6—C4	1.319 (14)
K6—O7^{ii}	2.817 (11)	F6—K14^{iii}	3.34 (6)
K6—O8^{ii}	3.090 (11)	F9—C6	1.300 (16)
K6—O1^{ii}	3.046 (12)	F9—K9^{vi}	3.33 (6)
K6—O10^{ii}	3.052 (11)	F3—C1	1.313 (12)
K8—K7^{i}	4.491 (6)	O12—C12	1.263 (13)
K8—K7	4.480 (5)	O12—K10	2.47 (6)
K8—O2	2.726 (11)	O4—C3	1.271 (15)
K8—F14^{iii}	3.057 (12)	O4—K12	2.53 (6)
K8—O8	2.698 (11)	F1—C1	1.296 (16)
K8—O4	2.791 (12)	F1—K12^{x}	3.29 (5)
K8—F5^{i}	2.920 (13)	O6—C5	1.252 (14)
K8—N7^{iv}	3.196 (18)	O6—K14^{ii}	2.59 (6)
K8—N3^{iv}	2.928 (15)	O10—C9	1.249 (15)
K8—N8^{iv}	2.942 (14)	O10—K13	2.49 (6)
K7—O5	2.728 (10)	F17—C11	1.299 (17)
K7—F12^{ii}	3.334 (14)	F17—K13^{xi}	3.40 (6)
K7—O8^{ii}	2.776 (12)	F2—C1	1.363 (16)
K7—O4	2.705 (12)	F2—K10^{ii}	2.29 (4)
K7—F1^{v}	3.118 (14)	F13—C10	1.379 (15)
K7—N2^{v}	2.941 (16)	F13—K14^{ii}	2.26 (4)
K7—N7^{iv}	2.897 (15)	F11—C7	1.370 (18)
K7—N3^{v}	3.210 (17)	F16—C11	1.344 (17)
K4—K3	4.464 (5)	F8—C6	1.309 (17)
K4—K3^{ii}	4.505 (5)	F8—K13^{ii}	2.38 (6)

K4—O7	2.724 (9)	C9—C10	1.5180 (2)
K4—O6	2.786 (13)	F5—C4	1.373 (16)
K4—O10	2.713 (12)	F5—K11^{ii}	2.28 (4)
K4—F8^{i}	2.992 (15)	C8—C7	1.5180 (2)
K4—N9^{vi}	2.963 (16)	C5—C6	1.5180 (2)
K4—N10^{vi}	3.302 (18)	C3—C4	1.5180 (2)
K4—N4^{vi}	3.148 (16)	N6—N10	1.27 (2)
K4—N5^{vi}	2.910 (16)	N6—N7	1.30 (2)
K3—O11	2.751 (11)	N6—K10	2.44 (7)
K3—F6^{vii}	3.194 (14)	N6—K12^{ix}	3.42 (6)
K3—O6^{i}	2.742 (11)	C2—C1	1.5180 (2)
K3—O10	2.793 (12)	N1—N2	1.29 (2)
K3—F13^{i}	2.914 (12)	N1—N5	1.28 (2)
K3—N9^{vi}	2.964 (17)	N1—K9	2.48 (7)
K3—N4^{viii}	2.931 (17)	C12—C11	1.5180 (2)
K3—N8^{vi}	3.006 (15)	N9—N10	1.29 (2)
K2—K1	4.469 (5)	N9—N8	1.317 (19)
K2—K1^{i}	4.499 (5)	N9—K13^{xi}	2.42 (6)
K2—O3^{i}	2.745 (11)	N2—N3	1.33 (2)
K2—F12^{ix}	3.082 (11)	C11—F16A	1.36 (2)
K2—F4^{i}	3.204 (11)	N10—K9^{i}	3.47 (6)
K2—O1	2.762 (12)	N10—K13^{xi}	2.86 (6)
K2—O12	2.711 (11)	N4—N5	1.28 (2)
K2—F2^{i}	2.912 (13)	N4—N3	1.29 (2)
K2—N6	2.908 (15)	N4—K11^{ix}	3.47 (6)
K2—N1	3.021 (16)	N4—K14^{xii}	2.45 (6)
K1—O9	2.725 (11)	N5—K13^{xi}	3.38 (5)
K1—O1	2.747 (11)	N5—K14^{xii}	3.17 (6)
K1—O12^{ii}	2.798 (13)	N7—N8	1.32 (2)
K1—F17^{ii}	3.150 (14)	N7—K12^{ix}	2.49 (5)
K1—N6^{ii}	3.180 (17)	N3—K11^{ix}	2.57 (6)
K1—N1	2.923 (16)	N3—K14^{xii}	3.42 (6)

K1—N10ii	2.920 (17)	N8—K12ix	3.44 (5)
O3—C3	1.214 (12)	N8—K14xi	3.44 (5)
O3—K10ii	2.83 (8)	K9—K10	4.469 (5)
O9—C9	1.242 (12)	K9—K10ii	4.48 (7)
O9—K9	2.82 (8)	K10—K12ix	4.64 (8)
O2—C2	1.231 (13)	K12—K11	4.480 (6)
O2—K11	2.70 (6)	K12—K11ii	4.50 (8)
O11—C12	1.221 (13)	K11—K14xiii	4.66 (7)
O11—K14	2.85 (6)	K13—K14ii	4.47 (8)
O5—C5	1.224 (14)	K13—K14	4.4630 (14)
O5—K12	2.77 (6)	K13—F16Avi	3.35 (6)
O7—C8	1.234 (14)		

Symmetry code(s): (i) *x*, *y*-1, *z*; (ii) *x*, *y*+1, *z*; (iii) *x*+1/2, -*y*+3/2, *z*; (iv) -*x*+1, -*y*+1, *z*-1/2; (v) -*x*+1, -*y*+2, *z*-1/2; (vi) -*x*+1/2, *y*+1/2, *z*-1/2; (vii) *x*-1/2, -*y*+3/2, *z*; (viii) -*x*+1/2, *y*-1/2, *z*-1/2; (ix) -*x*+1, -*y*+1, *z*+1/2; (x) -*x*+1, -*y*+2, *z*+1/2; (xi) -*x*+1/2, *y*-1/2, *z*+1/2; (xii) -*x*+1/2, *y*+1/2, *z*+1/2; (xiii) *x*+1/2, -*y*+1/2, *z*.

Table S6 Torsion angles [°] of **2**.

K5—O3—C3—O4	16.9 (15)	O2—C2—C1—F2	92.9 (12)
K5—O3—C3—C4	−155.5 (9)	O11—C12—C11—F18	14.3 (17)
K5—O9—C9—O10	66.9 (16)	O11—C12—C11—F17	134.1 (13)
K5—O9—C9—C10	−121.6 (10)	O11—C12—C11—F16	−100.1 (18)
K5—O2—C2—O1	69.8 (16)	O11—C12—C11—F16A	−123 (2)
K5—O2—C2—C1	−122.9 (9)	O5—C5—C6—F7	16.6 (16)
K5i—O11—C12—O12	18.0 (14)	O5—C5—C6—F9	138.6 (14)
K5i—O11—C12—C11	−155.5 (9)	O5—C5—C6—F8	−104.5 (14)
K5—O5—C5—O6	16.0 (15)	O7—C8—C7—F10	−23.9 (18)
K5—O5—C5—C6	−156.8 (8)	O7—C8—C7—F12	−151.3 (15)
K5—O7—C8—O8	68.8 (16)	O7—C8—C7—F11	93.5 (15)
K5—O7—C8—C7	−123.7 (10)	O8—C8—C7—F10	145.2 (13)
K5i—O12—C12—O11	−16.7 (13)	O8—C8—C7—F12	17.8 (18)
K5i—O12—C12—C11	157.2 (8)	O8—C8—C7—F11	−97.4 (15)
K5—O4—C3—O3	−15.4 (14)	F18—C11—F16A—K13v	112 (2)
K5—O4—C3—C4	157.3 (9)	O1—C2—C1—F3	144.0 (12)
K5—O6—C5—O5	−14.9 (14)	O1—C2—C1—F1	18.3 (16)
K5—O6—C5—C6	158.0 (9)	O1—C2—C1—F2	−98.3 (13)

K6—O3—C3—O4	−66.6 (16)	O12—C12—C11—F18	−159.9 (12)
K6—O3—C3—C4	121.0 (10)	O12—C12—C11—F17	−40.2 (14)
K6i—O9—C9—O10	−15.9 (15)	O12—C12—C11—F16	85.7 (19)
K6i—O9—C9—C10	155.6 (9)	O12—C12—C11—F16A	62 (2)
K6i—O2—C2—O1	−12.2 (14)	O4—C3—C4—F4	−155.4 (13)
K6i—O2—C2—C1	155.1 (9)	O4—C3—C4—F6	−29.1 (17)
K6i—O11—C12—O12	−65.9 (16)	O4—C3—C4—F5	87.3 (14)
K6i—O11—C12—C11	120.6 (9)	O6—C5—C6—F7	−157.1 (13)
K6—O5—C5—O6	−66.9 (16)	O6—C5—C6—F9	−35.1 (16)
K6—O5—C5—C6	120.4 (9)	O6—C5—C6—F8	81.8 (15)
K6i—O7—C8—O8	−14.0 (15)	O10—C9—C10—F15	141.4 (12)
K6i—O7—C8—C7	153.5 (9)	O10—C9—C10—F14	16.5 (18)
K6i—O8—C8—O7	12.6 (14)	O10—C9—C10—F13	−102.1 (14)
K6i—O8—C8—C7	−155.2 (10)	F17—C11—F16A—K13v	6 (2)
K6i—O1—C2—O2	11.2 (13)	N6—N7—N8—K8iv	−129.9 (12)
K6i—O1—C2—C1	−156.3 (9)	N6—N7—N8—K3v	94 (2)
K6i—O10—C9—O9	14.4 (14)	N6—N7—N8—N9	2.2 (19)
K6i—O10—C9—C10	−157.2 (10)	N6—N7—N8—K12iv	165 (2)
K8—O2—C2—O1	−139.7 (10)	N6—N7—N8—K14v	100 (2)
K8—O2—C2—C1	27.5 (18)	N1—N2—N3—K8iv	146.0 (12)
K8iii—F14—C10—F15	112.1 (14)	N1—N2—N3—K7vi	−135.1 (15)
K8iii—F14—C10—F13	−0.4 (17)	N1—N2—N3—N4	−1 (2)
K8iii—F14—C10—C9	−119.8 (13)	N1—N2—N3—K11iv	164.4 (17)
K8—O8—C8—O7	−90.0 (15)	N1—N2—N3—K14viii	−28 (2)
K8—O8—C8—C7	102.2 (11)	C12—C11—F16A—K13v	−110 (3)
K8—O4—C3—O3	−111.1 (13)	N2—N1—N5—K4v	−105.7 (15)
K8—O4—C3—C4	61.7 (13)	N2—N1—N5—N4	0 (2)
K8ii—F5—C4—F4	−102.0 (16)	N2—N1—N5—K13v	−96.5 (18)
K8ii—F5—C4—F6	144.4 (14)	N2—N1—N5—K14viii	39 (3)
K8ii—F5—C4—C3	21 (2)	N10—N6—N7—K8iv	−76.9 (19)
K8iv—N7—N8—N9	132.1 (14)	N10—N6—N7—K7iv	142.3 (13)
K7—O5—C5—O6	139.8 (11)	N10—N6—N7—N8	−4 (2)
K7—O5—C5—C6	−33.0 (15)	N10—N6—N7—K12iv	161.0 (19)
K7i—F12—C7—F10	−111.3 (9)	N10—N9—N8—K8iv	103.8 (16)
K7i—F12—C7—F11	135.1 (8)	N10—N9—N8—K3v	−149.2 (14)
K7i—F12—C7—C8	18.8 (15)	N10—N9—N8—N7	0 (2)
K7i—O8—C8—O7	107.8 (13)	N10—N9—N8—K12iv	−17 (3)
K7i—O8—C8—C7	−59.9 (13)	N10—N9—N8—K14v	−138.3 (17)
K7—O4—C3—O3	88.0 (15)	N5—N1—N2—K7vi	−104.9 (16)

K7—O4—C3—C4	−99.2 (11)	N5—N1—N2—N3	1 (2)
K7vi—F1—C1—F3	112.2 (12)	N5—N4—N3—K8iv	−146.7 (13)
K7vi—F1—C1—F2	−1.4 (12)	N5—N4—N3—K7vi	75 (2)
K7vi—F1—C1—C2	−119.0 (11)	N5—N4—N3—N2	1 (2)
K7vi—N2—N3—K8iv	−78.8 (10)	N5—N4—N3—K11iv	−164.5 (17)
K7vi—N2—N3—N4	133.8 (14)	N5—N4—N3—K14viii	143 (3)
K7iv—N7—N8—K8iv	81.9 (9)	N7—N6—N10—K4v	75 (3)
K7iv—N7—N8—K3v	−54 (2)	N7—N6—N10—K1i	−127.8 (13)
K7iv—N7—N8—N9	−145.9 (11)	N7—N6—N10—N9	4 (2)
K4—O7—C8—K6i	−128.7 (12)	N7—N6—N10—K9i	−117.3 (17)
K4—O7—C8—O8	−142.7 (11)	N7—N6—N10—K13v	52 (5)
K4—O7—C8—C7	24.8 (17)	N3—N4—N5—K4v	132.1 (14)
K4—O6—C5—K6	−155.6 (5)	N3—N4—N5—N1	−1 (2)
K4—O6—C5—O5	−114.3 (12)	N3—N4—N5—K13v	120.8 (17)
K4—O6—C5—C6	58.7 (13)	N3—N4—N5—K14viii	−150 (2)
K4—O10—C9—K6i	−104.6 (7)	N8—N9—N10—K4v	−151.5 (15)
K4—O10—C9—O9	−90.1 (15)	N8—N9—N10—K1i	104.4 (15)
K4—O10—C9—C10	98.2 (11)	N8—N9—N10—N6	−2 (2)
K4ii—F8—C6—F7	−86.2 (15)	N8—N9—N10—K9i	97.9 (16)
K4ii—F8—C6—F9	160.2 (12)	N8—N9—N10—K13v	−164 (2)
K4ii—F8—C6—C5	38 (2)	K9—O9—C9—O10	−123.5 (16)
K4v—N9—N10—K1i	−104.0 (11)	K9—O9—C9—C10	48.0 (18)
K4v—N9—N10—N6	149.3 (14)	K9—O1—C2—O2	−91 (3)
K4v—N9—N8—K8iv	−19 (3)	K9—O1—C2—C1	102 (3)
K4v—N9—N8—K3v	87.8 (18)	K9x—F9—C6—F7	−114.6 (18)
K4v—N9—N8—N7	−123.1 (18)	K9x—F9—C6—F8	0 (2)
K4v—N4—N5—N1	−133.3 (17)	K9x—F9—C6—C5	119.6 (18)
K4v—N4—N3—K8iv	−71.0 (18)	K9—N1—N2—N3	152 (3)
K4v—N4—N3—K7vi	151.1 (9)	K9—N1—N5—N4	−166.0 (18)
K4v—N4—N3—N2	77 (2)	K9—N1—N5—K13v	97.1 (19)
K3—O11—C12—K5i	124.6 (11)	K9—N1—N5—K14viii	−127 (2)
K3—O11—C12—K6i	−151.5 (14)	K10ii—O3—C3—O4	122.4 (18)
K3—O11—C12—O12	142.7 (10)	K10ii—O3—C3—C4	−50 (2)
K3—O11—C12—C11	−30.9 (17)	K10ix—F12—C7—F10	111.4 (19)
K3vii—F6—C4—F4	−112.1 (12)	K10ix—F12—C7—F11	−2 (2)
K3vii—F6—C4—F5	−0.9 (13)	K10ix—F12—C7—C8	−118.4 (19)
K3vii—F6—C4—C3	118.6 (10)	K10ii—F4—C4—F6	−118.3 (12)
K3ii—O6—C5—K6	49.4 (8)	K10ii—F4—C4—F5	131.2 (10)
K3ii—O6—C5—O5	90.6 (15)	K10ii—F4—C4—C3	11.5 (16)

K3ii—O6—C5—C6	−96.4 (11)	K10—O12—C12—O11	89 (3)
K3—O10—C9—O9	111.7 (12)	K10—O12—C12—C11	−97 (3)
K3—O10—C9—C10	−60.0 (14)	K10ii—F2—C1—F3	137 (3)
K3ii—F13—C10—F15	140.1 (14)	K10ii—F2—C1—F1	−109 (3)
K3ii—F13—C10—F14	−106.0 (15)	K10ii—F2—C1—C2	14 (4)
K3ii—F13—C10—C9	18.5 (19)	K10—N6—N10—N9	−155 (2)
K3v—N9—N10—K4v	112 (2)	K10—N6—N10—K9i	84 (2)
K3v—N9—N10—K1i	8 (3)	K10—N6—N10—K13v	−107 (5)
K3v—N9—N10—N6	−99 (2)	K10—N6—N7—N8	150.2 (19)
K3v—N9—N8—K8iv	−107.0 (11)	K10—N6—N7—K12iv	−45 (3)
K3v—N9—N8—N7	149.1 (13)	K12—O5—C5—O6	120.1 (16)
K3viii—N4—N5—K4v	−89.8 (7)	K12—O5—C5—C6	−52.7 (17)
K3viii—N4—N5—N1	136.9 (13)	K12—O4—C3—O3	82 (3)
K3viii—N4—N3—K8iv	87.3 (16)	K12—O4—C3—C4	−106 (3)
K3viii—N4—N3—K7vi	−51 (2)	K12vi—F1—C1—F3	109.0 (16)
K3viii—N4—N3—N2	−124.5 (14)	K12vi—F1—C1—F2	−4.6 (15)
K2ii—O3—C3—O4	143.1 (11)	K12vi—F1—C1—C2	−122.2 (15)
K2ii—O3—C3—C4	−29.4 (18)	K12iv—N6—N10—N9	22 (3)
K2ix—F12—C7—F10	113.4 (14)	K12iv—N6—N10—K9i	−99 (2)
K2ix—F12—C7—F11	−0.2 (17)	K12iv—N6—N10—K13v	70 (6)
K2ix—F12—C7—C8	−116.5 (12)	K12iv—N6—N7—N8	−165 (2)
K2ii—F4—C4—F6	−133.3 (10)	K12iv—N7—N8—N9	−162.5 (17)
K2ii—F4—C4—F5	116.1 (8)	K12iv—N7—N8—K14v	−65 (3)
K2ii—F4—C4—C3	−3.5 (15)	K11—O2—C2—O1	−117.2 (18)
K2—O1—C2—O2	109.4 (13)	K11—O2—C2—C1	50 (2)
K2—O1—C2—C1	−58.1 (13)	K11iii—F14—C10—F15	109.1 (17)
K2—O12—C12—O11	90.2 (14)	K11iii—F14—C10—F13	−3.5 (19)
K2—O12—C12—C11	−95.9 (11)	K11iii—F14—C10—C9	−122.8 (16)
K2ii—F2—C1—F3	123.7 (17)	K11—O8—C8—O7	−82 (3)
K2ii—F2—C1—F1	−121.7 (18)	K11—O8—C8—C7	110 (3)
K2ii—F2—C1—C2	1 (2)	K11ii—F5—C4—F4	−114 (3)
K2—N6—N10—K4v	−70 (3)	K11ii—F5—C4—F6	132 (3)
K2—N6—N10—K1i	87.5 (8)	K11ii—F5—C4—C3	9 (3)
K2—N6—N10—N9	−141.0 (13)	K11iv—N4—N5—N1	−17 (3)
K2—N6—N7—K8iv	58.6 (18)	K11iv—N4—N5—K13v	105 (2)
K2—N6—N7—K7iv	−82.2 (15)	K11iv—N4—N5—K14viii	−166 (3)
K2—N6—N7—N8	132.0 (12)	K11iv—N4—N3—N2	166 (3)
K2—N1—N2—K7vi	109.4 (13)	K11iv—N4—N3—K14viii	−52 (3)
K2—N1—N2—N3	−145.1 (14)	K13—O7—C8—O8	−122.3 (16)

K2—N1—N5—K4v	−12 (3)	K13—O7—C8—C7	45.2 (18)
K2—N1—N5—N4	94 (2)	K13—O10—C9—O9	−92 (3)
K1—O9—C9—O10	−143.4 (11)	K13—O10—C9—C10	96 (3)
K1—O9—C9—C10	28.1 (17)	K13v—F17—C11—F18	−118.1 (14)
K1—O1—C2—O2	−93.2 (14)	K13v—F17—C11—C12	119.3 (13)
K1—O1—C2—C1	99.3 (11)	K13v—F17—C11—F16A	−6 (2)
K1i—O12—C12—O11	−115.5 (12)	K13ii—F8—C6—F7	−104 (2)
K1i—O12—C12—C11	58.4 (12)	K13ii—F8—C6—F9	142 (2)
K1i—F17—C11—F18	130.1 (8)	K13ii—F8—C6—C5	20 (3)
K1i—F17—C11—F16	−113.5 (11)	K13v—N9—N10—N6	161.5 (19)
K1i—F17—C11—C12	7.4 (12)	K13v—N9—N10—K9i	−98.4 (17)
K1i—N6—N10—K4v	−157 (3)	K13v—N9—N8—N7	−144 (3)
K1i—N6—N10—N9	131.5 (17)	K13v—N9—N8—K12iv	−160 (3)
K1i—N6—N7—K8iv	−152.3 (7)	K13v—N9—N8—K14v	78 (4)
K1i—N6—N7—K7iv	66.9 (16)	K14—O11—C12—O12	124.0 (16)
K1i—N6—N7—N8	−78.9 (17)	K14—O11—C12—C11	−49.5 (19)
K1—N1—N2—K7vi	24 (3)	K14vii—F6—C4—F4	−108.6 (15)
K1—N1—N2—N3	129.8 (16)	K14vii—F6—C4—F5	2.6 (16)
K1—N1—N5—K4v	102.2 (13)	K14vii—F6—C4—C3	122.0 (14)
K1—N1—N5—N4	−151.8 (14)	K14ii—O6—C5—O5	88 (3)
O3—C3—C4—F4	18.1 (18)	K14ii—O6—C5—C6	−99 (2)
O3—C3—C4—F6	144.4 (13)	K14ii—F13—C10—F15	150 (3)
O3—C3—C4—F5	−99.2 (13)	K14ii—F13—C10—F14	−96 (3)
O9—C9—C10—F15	−31.0 (17)	K14ii—F13—C10—C9	29 (3)
O9—C9—C10—F14	−156.0 (13)	K14viii—N4—N5—N1	149.0 (18)
O9—C9—C10—F13	85.5 (13)	K14viii—N4—N5—K13v	−89.1 (17)
O2—C2—C1—F3	−24.8 (18)	K14viii—N4—N3—N2	−141.9 (18)
O2—C2—C1—F1	−150.5 (13)	K14viii—N4—N3—K11iv	52 (3)

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

2. Powder X-ray diffraction (PXRD) pattern

The powder pattern of a bulk sample of compounds **1** and **2**, and comparison with the pattern calculated from the single crystal data to prove the phase pure were shown in **Fig. S3**. It could be seen from **Fig.** S3a that the PXRD patterns of the experiment samples and calculated pattern were similar, and most of the peak positions were the same, with fewer miscellaneous peaks, which proved the purity of **1** was consistent with the experimental yield.

The peak positions of compound **2** were also mostly the same as the calculated values as shown in **Fig. S3**b, but there were relatively some miscellaneous peaks from 10 to 20 °, so the yield was slightly lower.

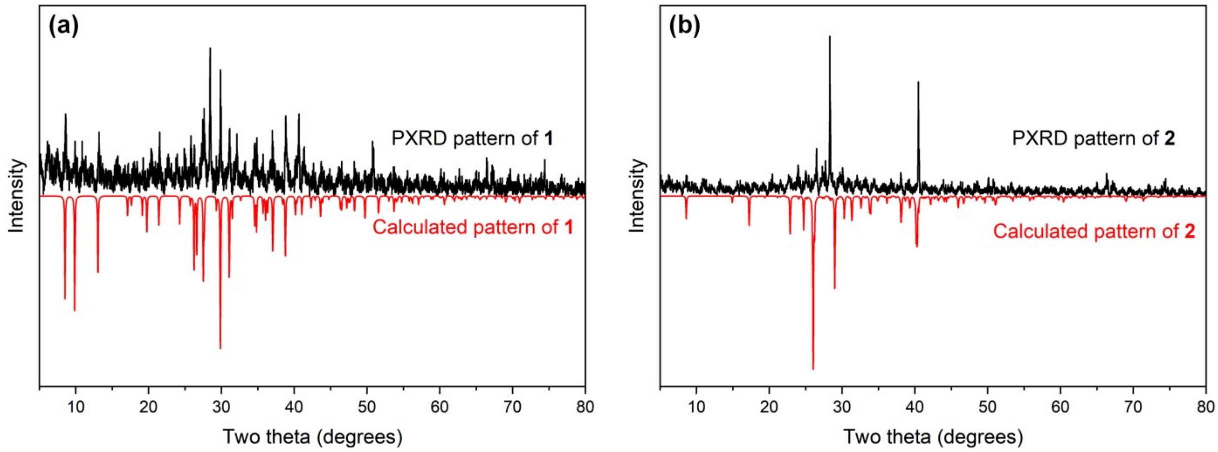

Fig. S3 (a, b) The PXRD pattern (in black) and the calculated pattern (in red) from their single-crystal structures of **1** and **2**.