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## Structure Reports

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**catena-Poly[[[diaquacopper(II)]- $\mu$ -pyridine-2,4-dicarboxylato- $\kappa^3$ N,O<sup>2</sup>:O<sup>4</sup>]  
0.625-hydrate]**

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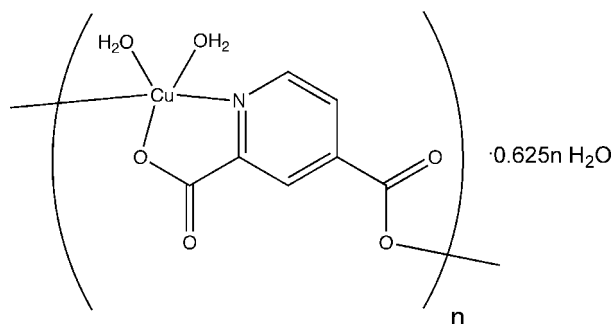
Received 15 November 2007; accepted 21 November 2007

Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å; disorder in solvent or counterion;  $R$  factor = 0.041;  $wR$  factor = 0.099; data-to-parameter ratio = 13.6.

In the title compound,  $[\text{Cu}(\text{C}_7\text{H}_3\text{NO}_4)(\text{H}_2\text{O})_2] \cdot 0.625\text{H}_2\text{O}$ , square-pyramidally coordinated  $\text{Cu}^{\text{II}}$  atoms with two *cis*-aqua ligands are linked into linear chains by bis-bridging pyridine-2,4-dicarboxylate (PDC) dianions. The  $[\text{Cu}(\text{PDC})(\text{H}_2\text{O})_2]_n$  chains form ribbons through long-range  $\text{Cu}-\text{O}$  interactions; the ribbons aggregate into a three-dimensional network through hydrogen bonding. The two uncoordinated water molecules of the asymmetric unit (one with partial occupancy) lie on different special positions of site symmetry 2.

## Related literature

For metal complexes of pyridinedicarboxylic acids, see: Zhou *et al.* (2006). For the synthesis of bis(3-pyridylmethyl)piperazine, see: Pocić *et al.* (2005).



## Experimental

## Crystal data

$[\text{Cu}(\text{C}_7\text{H}_3\text{NO}_4)(\text{H}_2\text{O})_2] \cdot 0.625\text{H}_2\text{O}$   
 $M_r = 275.94$

Orthorhombic,  $Pnna$   
 $a = 20.324$  (4) Å

$b = 12.057$  (2) Å  
 $c = 7.4706$  (16) Å  
 $V = 1830.7$  (6) Å<sup>3</sup>  
 $Z = 8$

Mo  $K\alpha$  radiation $\mu = 2.41$  mm<sup>-1</sup> $T = 173$  (2) K $0.25 \times 0.25 \times 0.20$  mm

## Data collection

Bruker APEXII diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\text{min}} = 0.520$ ,  $T_{\text{max}} = 0.618$

7418 measured reflections  
2152 independent reflections  
1517 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$  $wR(F^2) = 0.099$  $S = 1.09$ 

2152 reflections

158 parameters

21 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.68$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.69$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1W}-\text{H1W} \cdots \text{O2}^{\text{i}}$	0.86	2.65	3.145 (5)	117
$\text{O2W}-\text{H2W} \cdots \text{O1}^{\text{ii}}$	0.89	2.02	2.904 (17)	168
$\text{O5}-\text{H5A} \cdots \text{O1}^{\text{iii}}$	0.831 (18)	1.95 (2)	2.765 (4)	168 (4)
$\text{O5}-\text{H5B} \cdots \text{O1W}$	0.822 (18)	2.12 (2)	2.914 (4)	162 (4)
$\text{O6}-\text{H6A} \cdots \text{O4}^{\text{iv}}$	0.824 (18)	1.89 (2)	2.618 (4)	146 (4)
$\text{O6}-\text{H6B} \cdots \text{O2}^{\text{v}}$	0.815 (18)	2.02 (2)	2.719 (3)	144 (4)

Symmetry codes: (i)  $x, y, z - 1$ ; (ii)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ ; (iii)  $x, -y + \frac{3}{2}, -z + \frac{1}{2}$ ; (iv)  $-x + 1, y - \frac{1}{2}, z - \frac{1}{2}$ ; (v)  $-x + \frac{3}{2}, -y + 1, z$ .

Data collection: *COSMO* (Bruker, 2006); cell refinement: *APEX2* (Bruker, 2006); data reduction: *SAINT* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalMaker* (Palmer, 2005); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2377).

## References

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**supplementary materials**

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***catena*-Poly[[[diaquacopper(II)]- $\mu$ -pyridine-2,4-dicarboxylato- $\kappa^3N,O^2:O^4$ ] 0.625-hydrate]**

**D. P. Martin and R. L. LaDuca**

**Comment**

Coordination polymers containing both pyridinedicarboxylate and organodiiimine ligands have displayed intriguing structural morphologies and physical properties (Zhou *et al.*, 2006). The title compound was prepared during an attempt to prepare a divalent copper coordination polymer containing both 2,4-pyridinedicarboxylate (PDC) and *N,N'*-di(4-pyridylmethyl)piperazine (bpmp) ligands.

The asymmetric unit of the title compound (Fig. 1) consists of a Cu<sup>II</sup> ion, two aqua ligands, a single ligated PDC dianion, and waters molecule of crystallization located on crystallographic 2-fold axes (with 50% and 12.5% occupancies, respectively). The Cu<sup>II</sup> ion manifests a Jahn–Teller distorted square pyramidal coordination, with *cis* aqua ligands occupying the axial position and one of the basal positions. The remainder of the basal coordination sites are occupied by an O atom from one PDC ligand, and an N donor and O donor from a second PDC ligand. The two O donors from the PDC ligands, from 2- and 4-carboxylate positions, are situated *trans* to each other.

Neighboring Cu<sup>II</sup> ions are conjoined through exobidentate PDC ligands to form one-dimensional [Cu(H<sub>2</sub>O)<sub>2</sub>(PDC)]<sub>n</sub> chains that propagate along the [011] crystal direction (Fig. 2), with a Cu—Cu distance measuring 8.883 (3) Å. These are aggregated into double chain motifs (Fig. 3) through long-range Cu—O interactions (2.841 (2) Å).

Double [Cu(H<sub>2</sub>O)<sub>2</sub>(PDC)]<sub>n</sub> chains connect into *pseudo* layers, coincident with the *bc* crystal planes through hydrogen bonding between aqua ligands and unligated carboxylate O atoms. These subsequently stack into the *pseudo* three-dimensional crystal structure of the title compound through additional hydrogen bonding patterns involving the water molecules of crystallization (Fig. 4).

**Experimental**

Copper chloride dihydrate and 2,4-pyridinedicarboxylic acid (H<sub>2</sub>PDC) were obtained commercially. *N,N'*-di(4-pyridylmethyl)piperazine (bpmp) was prepared *via* a published procedure (Pocic *et al.*, 2005). Copper chloride dihydrate (0.044 g, 0.26 mmol) and H<sub>2</sub>PDC (0.031 g, 0.19 mmol) were dissolved in 4 ml water in a glass vial. A solution of bpmp (0.050 g, 0.19 mmol) in 4 ml *me* thanol was carefully layered on top. Blue blocks of the title compound were deposited (0.047 g, 0.17 mmol, 89% yield) after one week at 293 K.

**Refinement**

All H atoms bound to C atoms were placed in calculated positions, with C—H = 0.95 (2) Å and refined in riding mode with  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ . The H atoms bound to O in the aqua ligands and unligated water molecules were found *via* Fourier difference map, restrained with O—H = 0.85 (2) Å, and refined with  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{O})$ .

## Figures



Fig. 1. Asymmetric unit of the title compound, showing 50% probability ellipsoids and atom numbering scheme. Hydrogen atoms positions are marked as gray sticks. The unligated water molecule is omitted.



Fig. 2. A  $[\text{Cu}(\text{H}_2\text{O})_2(\text{PDC})]_n$  chain motif within the title compound. Color codes: light-blue N, black C, red O, dark blue Cu.

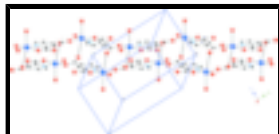


Fig. 3. A  $[\text{Cu}(\text{H}_2\text{O})_2(\text{PDC})]_{2n}$  double chain motif within the title compound. Long Cu—O interactions are shown as dashed lines.

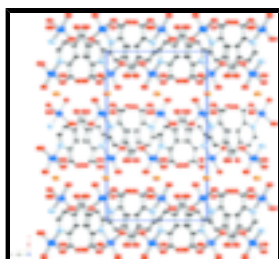


Fig. 4. Partial stacking diagram of the *pseudo* three-dimensional structure of the title compound. Unligated water molecules are shown in orange.

## catena-Poly[[[diaquacopper(II)]- $\mu$ -pyridine-2,4-dicarboxylato- $\kappa^3\text{N},\text{O}^2:\text{O}^4$ ] 0.625-hydrate]

### Crystal data

$[\text{Cu}(\text{C}_7\text{H}_3\text{N}_1\text{O}_4)(\text{H}_2\text{O})_2] \cdot 0.625\text{H}_2\text{O}$

$M_r = 275.94$

Orthorhombic,  $Pnna$

$a = 20.324 (4) \text{ \AA}$

$b = 12.057 (2) \text{ \AA}$

$c = 7.4706 (16) \text{ \AA}$

$V = 1830.7 (6) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1114$

$D_x = 2.006 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 7418 reflections

$\theta = 2.0\text{--}28.3^\circ$

$\mu = 2.41 \text{ mm}^{-1}$

$T = 173 (2) \text{ K}$

Block, blue

$0.25 \times 0.25 \times 0.20 \text{ mm}$

### Data collection

Bruker APEXII  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173(2) \text{ K}$

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)

2152 independent reflections

1517 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\text{max}} = 28.3^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -16 \rightarrow 26$

$T_{\min} = 0.520$ ,  $T_{\max} = 0.618$   
7418 measured reflections

$k = -10 \rightarrow 15$   
 $l = -6 \rightarrow 9$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.2838P]$
$S = 1.09$	where $P = (F_o^2 + 2F_c^2)/3$
2152 reflections	$(\Delta/\sigma)_{\max} = 0.001$
158 parameters	$\Delta\rho_{\max} = 0.68 \text{ e } \text{\AA}^{-3}$
21 restraints	$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cu1	0.631799 (18)	0.45906 (4)	0.18172 (6)	0.01542 (15)	
O1	0.67326 (11)	0.7225 (2)	0.4821 (3)	0.0251 (7)	
O1W	0.7500	0.5000	-0.3147 (6)	0.104 (3)	
H1W	0.7590	0.5513	-0.3903	0.124*	
O2W	0.7243 (11)	0.2500	-0.2500	0.108 (10)	0.25
H2W	0.7513	0.2379	-0.1577	0.130*	0.25
O2	0.68258 (10)	0.5671 (2)	0.3219 (3)	0.0174 (6)	
O3	0.42732 (10)	0.8454 (2)	0.5840 (3)	0.0166 (6)	
O4	0.35590 (11)	0.7053 (2)	0.5432 (4)	0.0320 (8)	
O5	0.64138 (12)	0.5631 (2)	-0.0813 (4)	0.0231 (7)	
H5A	0.6535 (18)	0.6282 (18)	-0.066 (5)	0.028*	
H5B	0.6750 (13)	0.560 (3)	-0.142 (4)	0.028*	
O6	0.71189 (11)	0.3848 (2)	0.1122 (4)	0.0208 (6)	
H6A	0.7049 (18)	0.3176 (17)	0.106 (5)	0.025*	

## supplementary materials

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H6B	0.7394 (14)	0.371 (3)	0.188 (4)	0.025*
N1	0.55748 (12)	0.5464 (2)	0.2784 (4)	0.0128 (7)
C1	0.57573 (15)	0.6319 (3)	0.3852 (5)	0.0129 (8)
C2	0.53100 (15)	0.7009 (3)	0.4690 (5)	0.0136 (8)
H2	0.5454	0.7614	0.5407	0.016*
C3	0.46437 (15)	0.6796 (3)	0.4454 (5)	0.0130 (8)
C4	0.44555 (15)	0.5921 (3)	0.3370 (4)	0.0163 (8)
H4	0.4003	0.5766	0.3188	0.020*
C5	0.49278 (15)	0.5276 (3)	0.2556 (5)	0.0154 (8)
H5	0.4793	0.4679	0.1808	0.018*
C6	0.64987 (15)	0.6440 (3)	0.4010 (5)	0.0152 (8)
C7	0.41055 (15)	0.7489 (3)	0.5317 (5)	0.0176 (8)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cu1	0.01020 (19)	0.0141 (3)	0.0220 (3)	-0.00002 (17)	-0.00034 (17)	-0.0044 (2)
O1	0.0160 (11)	0.0209 (16)	0.0385 (17)	-0.0024 (11)	-0.0026 (11)	-0.0116 (14)
O1W	0.096 (4)	0.190 (7)	0.024 (3)	0.102 (5)	0.000	0.000
O2W	0.095 (18)	0.17 (3)	0.057 (15)	0.000	0.000	-0.005 (18)
O2	0.0103 (10)	0.0161 (15)	0.0258 (15)	0.0003 (9)	0.0015 (10)	-0.0046 (13)
O3	0.0138 (11)	0.0137 (15)	0.0222 (14)	0.0014 (10)	0.0023 (10)	-0.0046 (13)
O4	0.0158 (13)	0.0252 (17)	0.055 (2)	-0.0062 (11)	0.0115 (12)	-0.0181 (16)
O5	0.0194 (13)	0.0228 (17)	0.0270 (17)	-0.0053 (11)	0.0009 (11)	-0.0024 (15)
O6	0.0116 (11)	0.0175 (16)	0.0332 (17)	0.0034 (11)	-0.0046 (11)	-0.0050 (15)
N1	0.0119 (12)	0.0108 (17)	0.0157 (17)	0.0004 (11)	0.0002 (11)	-0.0010 (14)
C1	0.0133 (14)	0.011 (2)	0.0141 (19)	-0.0028 (14)	0.0008 (13)	0.0007 (17)
C2	0.0163 (15)	0.009 (2)	0.016 (2)	-0.0020 (13)	0.0008 (14)	-0.0042 (16)
C3	0.0148 (15)	0.010 (2)	0.014 (2)	0.0003 (13)	0.0032 (13)	0.0028 (16)
C4	0.0128 (15)	0.014 (2)	0.022 (2)	0.0014 (14)	-0.0013 (14)	-0.0012 (18)
C5	0.0155 (15)	0.014 (2)	0.0172 (19)	0.0002 (14)	-0.0013 (14)	-0.0052 (17)
C6	0.0127 (15)	0.013 (2)	0.020 (2)	-0.0002 (14)	-0.0018 (14)	0.0009 (19)
C7	0.0158 (16)	0.017 (2)	0.020 (2)	0.0030 (15)	0.0016 (14)	-0.0009 (19)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Cu1—O6	1.931 (2)	O6—H6A	0.824 (18)
Cu1—O2	1.963 (2)	O6—H6B	0.815 (18)
Cu1—O3 <sup>i</sup>	1.964 (2)	N1—C5	1.348 (4)
Cu1—N1	1.980 (3)	N1—C1	1.353 (4)
Cu1—O5	2.339 (3)	C1—C2	1.383 (4)
O1—C6	1.219 (4)	C1—C6	1.522 (4)
O1W—H1W	0.8563	C2—C3	1.392 (4)
O2W—H2W	0.8920	C2—H2	0.9500
O2—C6	1.285 (4)	C3—C4	1.382 (5)
O3—C7	1.273 (4)	C3—C7	1.521 (4)
O4—C7	1.234 (4)	C4—C5	1.378 (4)
O5—H5A	0.831 (18)	C4—H4	0.9500

O5—H5B	0.822 (18)	C5—H5	0.9500
O6—Cu1—O2	90.46 (10)	N1—C1—C2	122.8 (3)
O6—Cu1—O3 <sup>i</sup>	95.30 (11)	N1—C1—C6	113.0 (3)
O2—Cu1—O3 <sup>i</sup>	169.33 (10)	C2—C1—C6	124.1 (3)
O6—Cu1—N1	171.84 (11)	C1—C2—C3	118.3 (3)
O2—Cu1—N1	81.58 (10)	C1—C2—H2	120.8
O3 <sup>i</sup> —Cu1—N1	92.30 (10)	C3—C2—H2	120.8
O6—Cu1—O5	87.36 (11)	C4—C3—C2	119.0 (3)
O2—Cu1—O5	92.78 (9)	C4—C3—C7	117.8 (3)
O3 <sup>i</sup> —Cu1—O5	96.48 (10)	C2—C3—C7	123.2 (3)
N1—Cu1—O5	94.86 (11)	C5—C4—C3	119.6 (3)
C6—O2—Cu1	116.7 (2)	C5—C4—H4	120.2
C7—O3—Cu1 <sup>iii</sup>	125.9 (2)	C3—C4—H4	120.2
Cu1—O5—H5A	115 (3)	N1—C5—C4	122.1 (3)
Cu1—O5—H5B	120 (3)	N1—C5—H5	118.9
H5A—O5—H5B	83 (3)	C4—C5—H5	118.9
Cu1—O6—H6A	109 (3)	O1—C6—O2	125.8 (3)
Cu1—O6—H6B	119 (3)	O1—C6—C1	120.0 (3)
H6A—O6—H6B	87 (3)	O2—C6—C1	114.2 (3)
C5—N1—C1	118.1 (3)	O4—C7—O3	127.6 (3)
C5—N1—Cu1	127.7 (2)	O4—C7—C3	116.4 (3)
C1—N1—Cu1	114.1 (2)	O3—C7—C3	116.0 (3)
O6—Cu1—O2—C6	178.6 (3)	C2—C3—C4—C5	0.4 (5)
O3 <sup>i</sup> —Cu1—O2—C6	-58.7 (7)	C7—C3—C4—C5	179.7 (3)
N1—Cu1—O2—C6	-3.2 (3)	C1—N1—C5—C4	-0.1 (5)
O5—Cu1—O2—C6	91.2 (3)	Cu1—N1—C5—C4	177.0 (3)
O2—Cu1—N1—C5	-176.9 (3)	C3—C4—C5—N1	0.3 (6)
O3 <sup>i</sup> —Cu1—N1—C5	-5.7 (3)	Cu1—O2—C6—O1	-174.1 (3)
O5—Cu1—N1—C5	91.1 (3)	Cu1—O2—C6—C1	5.1 (4)
O2—Cu1—N1—C1	0.3 (2)	N1—C1—C6—O1	174.5 (3)
O3 <sup>i</sup> —Cu1—N1—C1	171.5 (2)	C2—C1—C6—O1	-4.9 (6)
O5—Cu1—N1—C1	-91.7 (2)	N1—C1—C6—O2	-4.8 (5)
C5—N1—C1—C2	-0.9 (5)	C2—C1—C6—O2	175.8 (3)
Cu1—N1—C1—C2	-178.4 (3)	Cu1 <sup>ii</sup> —O3—C7—O4	-5.5 (6)
C5—N1—C1—C6	179.7 (3)	Cu1 <sup>ii</sup> —O3—C7—C3	174.6 (2)
Cu1—N1—C1—C6	2.2 (4)	C4—C3—C7—O4	20.4 (5)
N1—C1—C2—C3	1.6 (5)	C2—C3—C7—O4	-160.3 (4)
C6—C1—C2—C3	-179.1 (3)	C4—C3—C7—O3	-159.7 (3)
C1—C2—C3—C4	-1.3 (5)	C2—C3—C7—O3	19.6 (5)
C1—C2—C3—C7	179.4 (3)		

Symmetry codes: (i)  $-x+1, y-1/2, z-1/2$ ; (ii)  $-x+1, y+1/2, z+1/2$ .

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W $\cdots$ O2 <sup>iii</sup>	0.86	2.65	3.145 (5)	117

## supplementary materials

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O2W—H2W...O1 <sup>iv</sup>	0.89	2.02	2.904 (17)	168
O5—H5A...O1 <sup>v</sup>	0.831 (18)	1.95 (2)	2.765 (4)	168 (4)
O5—H5B...O1W	0.822 (18)	2.12 (2)	2.914 (4)	162 (4)
O6—H6A...O4 <sup>i</sup>	0.824 (18)	1.89 (2)	2.618 (4)	146 (4)
O6—H6B...O2 <sup>vi</sup>	0.815 (18)	2.02 (2)	2.719 (3)	144 (4)

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



Fig. 1

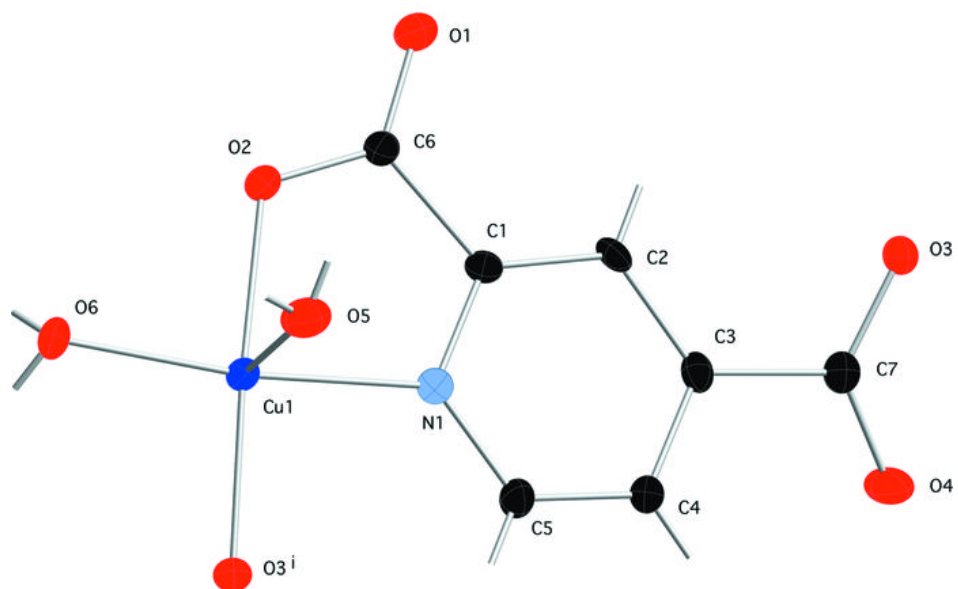


Fig. 2

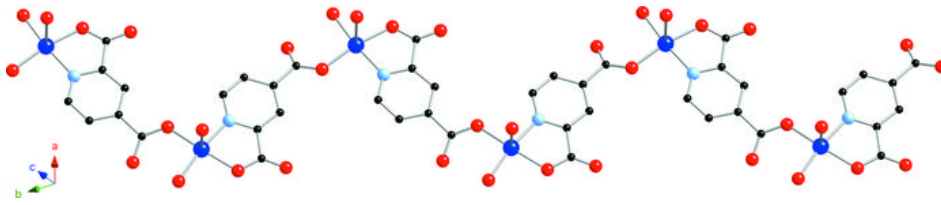


Fig. 3

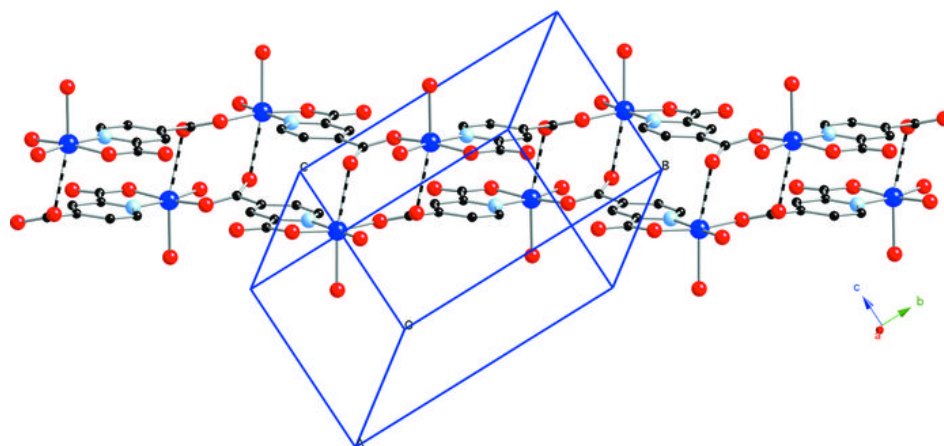


Fig. 4

