

Poly[bis(2,2'-bipyridine- κ^2N,N')deca- μ -oxido-dioxidodicopper(II)tetra-vanadium(V)]

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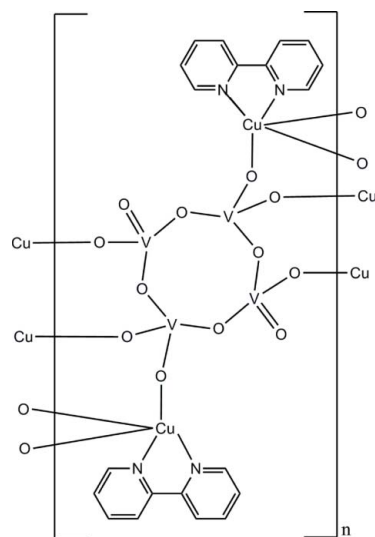
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.034; wR factor = 0.084; data-to-parameter ratio = 16.4.

The title compound, $[\text{Cu}_2\text{V}_4\text{O}_{12}(\text{C}_{10}\text{H}_8\text{N}_2)_2]_n$, shows a two-dimensional copper–vanadate layer composed of eight-membered rings, each containing four corner-sharing VO_4 tetrahedra; these are linked through six pentacoordinated Cu^{II} atoms with the 2,2'-bipyridine ligands attached and pointing above and below the plane of the layer. The Cu atom is coordinated by two N donors from the 2,2'-bipyridine ligand and three O atoms from three adjacent VO_4 units to form a distorted tetragonal pyramid. These layers are further connected by π – π interactions between interleaving bipyridine ligands of adjacent layers [centroid–centroid distances = 3.63 (1) and 3.68 (1) Å] into a three-dimensional supramolecular structure.

Related literature

For related literature, see: DeBord *et al.* (1996); Kucsera *et al.* (2002); Lu *et al.* (2002); Yi *et al.* (2007).



Experimental

Crystal data

$[\text{Cu}_2\text{V}_4\text{O}_{12}(\text{C}_{10}\text{H}_8\text{N}_2)_2]$
 $M_r = 417.60$
 Triclinic, $P\bar{1}$
 $a = 8.1019$ (4) Å
 $b = 8.3122$ (5) Å
 $c = 10.3501$ (4) Å
 $\alpha = 72.332$ (3)°
 $\beta = 84.562$ (3)°

$\gamma = 77.878$ (3)°
 $V = 648.98$ (6) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 3.06$ mm⁻¹
 $T = 298$ (2) K
 $0.33 \times 0.31 \times 0.25$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\text{min}} = 0.379$, $T_{\text{max}} = 0.469$

4603 measured reflections
 3114 independent reflections
 2553 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.084$
 $S = 0.99$
 3114 reflections

190 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.75$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cu1–O1	2.012 (2)	V1–O5 ⁱⁱⁱ	1.824 (2)
Cu1–O4 ⁱ	2.054 (2)	V1–O3	1.833 (2)
Cu1–O6 ⁱⁱ	2.061 (2)	V2–O4	1.655 (2)
Cu1–N1	2.084 (2)	V2–O6	1.670 (2)
Cu1–N2	2.117 (2)	V2–O5	1.774 (2)
V1–O2	1.615 (2)	V2–O3	1.790 (2)
V1–O1	1.667 (2)		
O1–Cu1–O4 ⁱ	89.62 (9)	O6 ⁱⁱ –Cu1–N2	93.05 (9)
O1–Cu1–O6 ⁱⁱ	94.53 (9)	N1–Cu1–N2	78.18 (10)
O4 ⁱ –Cu1–O6 ⁱⁱ	121.32 (9)	O2–V1–O1	108.65 (12)
O1–Cu1–N1	100.17 (9)	O2–V1–O5 ⁱⁱⁱ	109.43 (12)
O4 ⁱ –Cu1–N1	124.55 (9)	O1–V1–O5 ⁱⁱⁱ	111.53 (11)
O6 ⁱⁱ –Cu1–N1	112.18 (9)	O2–V1–O3	107.98 (12)
O1–Cu1–N2	172.29 (9)	O1–V1–O3	110.08 (11)
O4 ⁱ –Cu1–N2	85.25 (9)	O5 ⁱⁱⁱ –V1–O3	109.09 (10)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); 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: HY2113).

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supplementary materials

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Poly[bis(2,2'-bipyridine- κ^2N,N')deca- μ -oxido-dioxidodicopper(II)tetravanadium(V)]

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Comment

Considerable efforts have been devoted to the hydrothermal synthesis of solid-state inorganic–organic hybrid vanadate(V) species based on discrete clusters, infinite chain and layer structures, such as [Zn(phen)₃][V₂O₆].10H₂O and [Cu(bipy)V₂O₆] (Yi *et al.*, 2007), [Cu(bipy)][V₂O₆] and [Cu(bipy)₂][V₂O₆] (DeBord *et al.*, 1996), [Mn(phen)₂]₂[V₄O₁₂].0.5H₂O (Lu *et al.*, 2002), and [Co(phen)₂]₂[V₄O₁₂].H₂O (Kucsera *et al.*, 2002), because of their diverse topologies and fascinating physical properties. We report here the crystal structure of a new complex, {[Cu(bipy)]₂V₄O₁₂}_n (bipy = 2,2'-bipyridine).

The asymmetric unit of the title compound consists of one Cu^{II} atom, one bipy molecule and a half of V₄O₁₂ unit (Fig. 1). The V₄O₁₂ units are linked through six square-pyramidal Cu^{II} atoms to six adjacent V₄O₁₂ rings (Fig. 2). Two of VO₄ units in the V₄O₁₂ unit each connect with one square-pyramidal Cu unit, while the other two VO₄ units each exhibit corner-sharing interactions with two Cu units. Each Cu unit links three V₄O₁₂ units through corner-sharing interactions. In this way, a two-dimensional layer is formed (Fig. 2). The Cu^{II} atom is coordinated by two pyridine N atoms and three tetravanadate O atoms (Fig. 1 and Table 1). The relative orientation of the bipy ligand with respect to the copper–vanadate layer is depicted by a dihedral angle of 84.6 (6)°. Furthermore, these bipy ligands interact with each other through π – π interactions between adjacent layers with centroid–centroid distances of 3.63 (1) and 3.68 (1) Å.

Experimental

The title compound was prepared hydrothermally from a mixture of V₂O₅ (0.73 g, 4.0 mmol), 2,2'-bipyridine dihydrate (0.38 g, 2.0 mmol), CuCl₂.2H₂O (0.34 g, 2.0 mmol) and water (18 ml) (molar ratio 2:1:1:500), adjusting pH to *ca* 6.1 with 4 M KOH, in a 25 ml Teflon-lined stainless steel reactor heated to 443 K for 7 d. After cooling to room temperature, green crystals were collected.

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

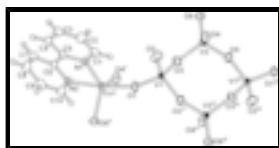


Fig. 1. The asymmetric unit of the title compound, extended to show the V₄O₁₂ unit. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) 1 - x, 1 - y, 1 - z; (ii) x, y - 1, z; (iii) 2 - x, 1 - y, 1 - z.]

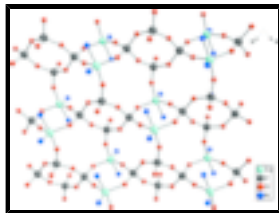


Fig. 2. A view of the copper–vanadate layer with C and H atoms of the bipy ligands omitted for clarity.

poly[bis(2,2'-bipyridine- κ^2N,N')deca- μ -oxido- dioxidodicopper(II)tetravanadium(V)],

Crystal data

[Cu₂V₄O₁₂(C₁₀H₈N₂)₂]

$M_r = 417.60$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.1019$ (4) Å

$b = 8.3122$ (5) Å

$c = 10.3501$ (4) Å

$\alpha = 72.332$ (3)°

$\beta = 84.562$ (3)°

$\gamma = 77.878$ (3)°

$V = 648.98$ (6) Å³

$Z = 2$

$F_{000} = 410$

$D_x = 2.137$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3811 reflections

$\theta = 2.1$ – 28.3 °

$\mu = 3.06$ mm⁻¹

$T = 298$ (2) K

Block, green

$0.33 \times 0.31 \times 0.25$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2002)

$T_{\min} = 0.379$, $T_{\max} = 0.469$

4603 measured reflections

3114 independent reflections

2553 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\text{max}} = 28.3$ °

$\theta_{\text{min}} = 2.1$ °

$h = -10 \rightarrow 9$

$k = -10 \rightarrow 7$

$l = -13 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.084$

$S = 0.99$

3114 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.53$ e Å⁻³

190 parameters

$$\Delta\rho_{\min} = -0.75 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.59216 (4)	0.14236 (4)	0.28662 (3)	0.01368 (10)
V1	0.87679 (6)	0.39002 (6)	0.34998 (5)	0.01604 (12)
V2	0.73879 (6)	0.72867 (6)	0.47736 (5)	0.01495 (12)
O1	0.7741 (3)	0.2421 (3)	0.3367 (2)	0.0249 (5)
O2	0.9198 (3)	0.5062 (3)	0.1993 (2)	0.0359 (6)
O3	0.7425 (3)	0.5317 (3)	0.4394 (2)	0.0259 (5)
O4	0.5705 (3)	0.7685 (3)	0.5748 (2)	0.0270 (5)
O5	0.9278 (3)	0.7084 (3)	0.5595 (2)	0.0285 (5)
O6	0.7286 (3)	0.8942 (3)	0.3363 (2)	0.0247 (5)
N1	0.5900 (3)	0.2590 (3)	0.0777 (2)	0.0191 (5)
N2	0.3794 (3)	0.0700 (3)	0.2320 (2)	0.0202 (5)
C1	0.7065 (4)	0.3461 (4)	0.0041 (3)	0.0242 (6)
H1	0.7903	0.3671	0.0489	0.029*
C2	0.7057 (4)	0.4056 (4)	-0.1364 (3)	0.0300 (7)
H2	0.7869	0.4667	-0.1848	0.036*
C3	0.5828 (4)	0.3731 (4)	-0.2037 (3)	0.0270 (7)
H3	0.5811	0.4107	-0.2980	0.032*
C4	0.4620 (4)	0.2835 (4)	-0.1289 (3)	0.0232 (6)
H4	0.3787	0.2594	-0.1722	0.028*
C5	0.4675 (4)	0.2303 (4)	0.0120 (3)	0.0192 (6)
C6	0.3397 (4)	0.1390 (4)	0.1003 (3)	0.0187 (6)
C7	0.1878 (4)	0.1270 (4)	0.0534 (3)	0.0288 (7)
H7	0.1610	0.1775	-0.0372	0.035*
C8	0.0781 (4)	0.0386 (4)	0.1448 (4)	0.0309 (7)
H8	-0.0237	0.0290	0.1160	0.037*
C9	0.1209 (4)	-0.0351 (4)	0.2786 (3)	0.0274 (7)
H9	0.0492	-0.0962	0.3409	0.033*
C10	0.2718 (4)	-0.0166 (4)	0.3187 (3)	0.0247 (6)
H10	0.3001	-0.0660	0.4091	0.030*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.01353 (17)	0.01631 (18)	0.01260 (17)	-0.00306 (12)	-0.00204 (12)	-0.00563 (13)
V1	0.0140 (2)	0.0172 (2)	0.0183 (2)	-0.00322 (18)	-0.00335 (18)	-0.00613 (19)
V2	0.0127 (2)	0.0155 (2)	0.0177 (2)	-0.00107 (17)	-0.00215 (18)	-0.00696 (18)
O1	0.0202 (11)	0.0282 (12)	0.0335 (12)	-0.0079 (9)	-0.0034 (9)	-0.0167 (10)
O2	0.0332 (14)	0.0428 (14)	0.0249 (12)	-0.0122 (11)	-0.0004 (10)	0.0031 (11)
O3	0.0243 (12)	0.0225 (11)	0.0346 (13)	-0.0036 (9)	0.0016 (10)	-0.0154 (10)
O4	0.0201 (11)	0.0366 (13)	0.0301 (12)	-0.0050 (9)	0.0047 (9)	-0.0201 (10)
O5	0.0228 (12)	0.0308 (12)	0.0346 (13)	-0.0013 (9)	-0.0112 (10)	-0.0128 (10)

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O6	0.0245 (11)	0.0204 (11)	0.0266 (11)	-0.0018 (9)	-0.0051 (9)	-0.0034 (9)
N1	0.0195 (12)	0.0206 (12)	0.0179 (12)	-0.0040 (10)	-0.0029 (10)	-0.0059 (10)
N2	0.0225 (13)	0.0233 (13)	0.0178 (12)	-0.0065 (10)	-0.0011 (10)	-0.0088 (10)
C1	0.0211 (15)	0.0252 (16)	0.0269 (16)	-0.0057 (12)	-0.0012 (12)	-0.0077 (13)
C2	0.0264 (17)	0.0326 (18)	0.0299 (17)	-0.0117 (14)	0.0065 (14)	-0.0058 (14)
C3	0.0328 (18)	0.0269 (16)	0.0177 (15)	-0.0036 (13)	-0.0008 (13)	-0.0027 (12)
C4	0.0261 (16)	0.0256 (16)	0.0199 (15)	-0.0037 (12)	-0.0050 (12)	-0.0090 (12)
C5	0.0181 (14)	0.0190 (14)	0.0211 (14)	-0.0013 (11)	-0.0044 (11)	-0.0072 (11)
C6	0.0188 (14)	0.0207 (14)	0.0188 (14)	-0.0048 (11)	-0.0023 (11)	-0.0078 (11)
C7	0.0270 (17)	0.0350 (18)	0.0252 (16)	-0.0084 (14)	-0.0101 (13)	-0.0056 (14)
C8	0.0204 (16)	0.0381 (19)	0.0376 (19)	-0.0095 (14)	-0.0062 (14)	-0.0118 (15)
C9	0.0242 (16)	0.0299 (17)	0.0318 (18)	-0.0129 (13)	0.0058 (13)	-0.0110 (14)
C10	0.0263 (17)	0.0295 (17)	0.0192 (14)	-0.0094 (13)	0.0023 (12)	-0.0065 (12)

Geometric parameters (\AA , $^\circ$)

Cu1—O1	2.012 (2)	N2—C6	1.350 (4)
Cu1—O4 ⁱ	2.054 (2)	C1—C2	1.387 (4)
Cu1—O6 ⁱⁱ	2.061 (2)	C1—H1	0.9300
Cu1—N1	2.084 (2)	C2—C3	1.381 (5)
Cu1—N2	2.117 (2)	C2—H2	0.9300
V1—O2	1.615 (2)	C3—C4	1.388 (5)
V1—O1	1.667 (2)	C3—H3	0.9300
V1—O5 ⁱⁱⁱ	1.824 (2)	C4—C5	1.392 (4)
V1—O3	1.833 (2)	C4—H4	0.9300
V2—O4	1.655 (2)	C5—C6	1.488 (4)
V2—O6	1.670 (2)	C6—C7	1.397 (4)
V2—O5	1.774 (2)	C7—C8	1.384 (5)
V2—O3	1.790 (2)	C7—H7	0.9300
O4—Cu1 ⁱ	2.054 (2)	C8—C9	1.379 (5)
O5—V1 ⁱⁱⁱ	1.824 (2)	C8—H8	0.9300
O6—Cu1 ^{iv}	2.061 (2)	C9—C10	1.379 (4)
N1—C1	1.346 (4)	C9—H9	0.9300
N1—C5	1.352 (4)	C10—H10	0.9300
N2—C10	1.344 (4)		
O1—Cu1—O4 ⁱ	89.62 (9)	C6—N2—Cu1	114.9 (2)
O1—Cu1—O6 ⁱⁱ	94.53 (9)	N1—C1—C2	122.1 (3)
O4 ⁱ —Cu1—O6 ⁱⁱ	121.32 (9)	N1—C1—H1	118.9
O1—Cu1—N1	100.17 (9)	C2—C1—H1	118.9
O4 ⁱ —Cu1—N1	124.55 (9)	C3—C2—C1	119.2 (3)
O6 ⁱⁱ —Cu1—N1	112.18 (9)	C3—C2—H2	120.4
O1—Cu1—N2	172.29 (9)	C1—C2—H2	120.4
O4 ⁱ —Cu1—N2	85.25 (9)	C2—C3—C4	119.1 (3)
O6 ⁱⁱ —Cu1—N2	93.05 (9)	C2—C3—H3	120.4
N1—Cu1—N2	78.18 (10)	C4—C3—H3	120.4
O2—V1—O1	108.65 (12)	C3—C4—C5	118.9 (3)

O2—V1—O5 ⁱⁱⁱ	109.43 (12)	C3—C4—H4	120.6
O1—V1—O5 ⁱⁱⁱ	111.53 (11)	C5—C4—H4	120.6
O2—V1—O3	107.98 (12)	N1—C5—C4	121.9 (3)
O1—V1—O3	110.08 (11)	N1—C5—C6	115.6 (3)
O5 ⁱⁱⁱ —V1—O3	109.09 (10)	C4—C5—C6	122.5 (3)
O4—V2—O6	107.88 (11)	N2—C6—C7	121.6 (3)
O4—V2—O5	111.20 (11)	N2—C6—C5	114.8 (2)
O6—V2—O5	108.55 (11)	C7—C6—C5	123.6 (3)
O4—V2—O3	109.63 (11)	C8—C7—C6	118.6 (3)
O6—V2—O3	111.29 (11)	C8—C7—H7	120.7
O5—V2—O3	108.30 (10)	C6—C7—H7	120.7
V1—O1—Cu1	159.01 (14)	C9—C8—C7	119.6 (3)
V2—O3—V1	139.64 (14)	C9—C8—H8	120.2
V2—O4—Cu1 ⁱ	162.64 (14)	C7—C8—H8	120.2
V2—O5—V1 ⁱⁱⁱ	160.20 (14)	C8—C9—C10	118.9 (3)
V2—O6—Cu1 ^{iv}	133.16 (13)	C8—C9—H9	120.6
C1—N1—C5	118.7 (3)	C10—C9—H9	120.6
C1—N1—Cu1	125.5 (2)	N2—C10—C9	122.5 (3)
C5—N1—Cu1	115.6 (2)	N2—C10—H10	118.7
C10—N2—C6	118.8 (3)	C9—C10—H10	118.7
C10—N2—Cu1	125.7 (2)		
O2—V1—O1—Cu1	-60.3 (4)	N1—Cu1—N2—C10	-175.0 (3)
O5 ⁱⁱⁱ —V1—O1—Cu1	179.0 (4)	O4 ⁱ —Cu1—N2—C6	122.4 (2)
O3—V1—O1—Cu1	57.8 (4)	O6 ⁱⁱ —Cu1—N2—C6	-116.4 (2)
O4 ⁱ —Cu1—O1—V1	-68.8 (4)	N1—Cu1—N2—C6	-4.4 (2)
O6 ⁱⁱ —Cu1—O1—V1	169.8 (4)	C5—N1—C1—C2	-0.6 (5)
N1—Cu1—O1—V1	56.3 (4)	Cu1—N1—C1—C2	173.5 (2)
O4—V2—O3—V1	-176.89 (19)	N1—C1—C2—C3	-0.8 (5)
O6—V2—O3—V1	63.8 (2)	C1—C2—C3—C4	0.8 (5)
O5—V2—O3—V1	-55.4 (2)	C2—C3—C4—C5	0.5 (5)
O2—V1—O3—V2	-51.5 (2)	C1—N1—C5—C4	2.0 (4)
O1—V1—O3—V2	-169.94 (19)	Cu1—N1—C5—C4	-172.6 (2)
O5 ⁱⁱⁱ —V1—O3—V2	67.4 (2)	C1—N1—C5—C6	-177.8 (3)
O6—V2—O4—Cu1 ⁱ	-84.8 (5)	Cu1—N1—C5—C6	7.5 (3)
O5—V2—O4—Cu1 ⁱ	34.1 (5)	C3—C4—C5—N1	-2.0 (4)
O3—V2—O4—Cu1 ⁱ	153.8 (4)	C3—C4—C5—C6	177.9 (3)
O4—V2—O5—V1 ⁱⁱⁱ	104.6 (4)	C10—N2—C6—C7	2.1 (4)
O6—V2—O5—V1 ⁱⁱⁱ	-136.9 (4)	Cu1—N2—C6—C7	-169.2 (2)
O3—V2—O5—V1 ⁱⁱⁱ	-15.9 (5)	C10—N2—C6—C5	-179.3 (3)
O4—V2—O6—Cu1 ^{iv}	17.23 (19)	Cu1—N2—C6—C5	9.5 (3)
O5—V2—O6—Cu1 ^{iv}	-103.38 (17)	N1—C5—C6—N2	-11.3 (4)
O3—V2—O6—Cu1 ^{iv}	137.53 (15)	C4—C5—C6—N2	168.8 (3)
O1—Cu1—N1—C1	11.4 (3)	N1—C5—C6—C7	167.3 (3)
O4 ⁱ —Cu1—N1—C1	108.1 (2)	C4—C5—C6—C7	-12.5 (5)

supplementary materials

O6 ⁱⁱ —Cu1—N1—C1	-87.8 (3)	N2—C6—C7—C8	-1.5 (5)
N2—Cu1—N1—C1	-176.2 (3)	C5—C6—C7—C8	180.0 (3)
O1—Cu1—N1—C5	-174.30 (19)	C6—C7—C8—C9	0.0 (5)
O4 ⁱ —Cu1—N1—C5	-77.7 (2)	C7—C8—C9—C10	0.9 (5)
O6 ⁱⁱ —Cu1—N1—C5	86.5 (2)	C6—N2—C10—C9	-1.2 (5)
N2—Cu1—N1—C5	-1.97 (19)	Cu1—N2—C10—C9	169.1 (2)
O4 ⁱ —Cu1—N2—C10	-48.2 (3)	C8—C9—C10—N2	-0.3 (5)
O6 ⁱⁱ —Cu1—N2—C10	73.0 (3)		

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

Fig. 1

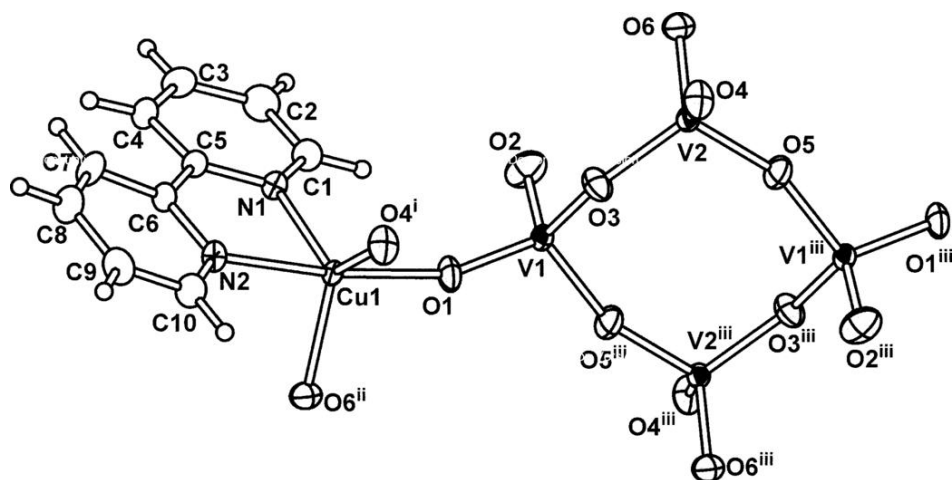


Fig. 2

