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

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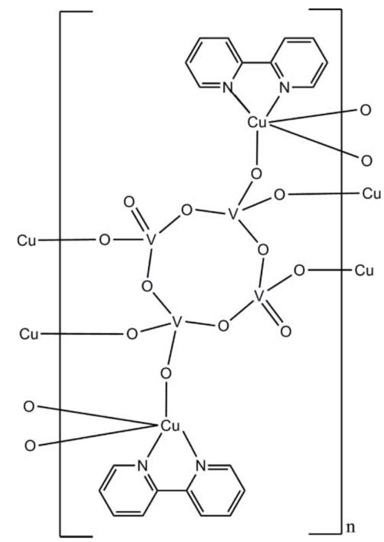
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.034; wR factor = 0.084; data-to-parameter ratio = 16.4.

The title compound, $[Cu_2V_4O_{12}(C_{10}H_8N_2)_2]_n$, 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

$[Cu_2V_4O_{12}(C_{10}H_8N_2)_2]$
M_r = 417.60
Triclinic, $P\overline{1}$
a = 8.1019 (4) Å
b = 8.3122 (5) Å
c = 10.3501 (4) Å
α = 72.332 (3)°
β = 84.562 (3)°
γ = 77.878 (3)°
V = 648.98 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.06 mm^{-1}
T = 298 (2) K
0.33 × 0.31 × 0.25 mm

Data collection

Bruker SMART APEX CCD diffractometer
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_{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
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −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: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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^2*N*,*N*')deca-μ-oxido-dioxidodicopper(II)tetravanadium(V)]

X. Zhang, W. Xia, X. Xu, Z. Yi and C. Yang

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)$_3$][V_2O_6].10H_2O and [Cu(bipy)V_2O_6] (Yi *et al.*, 2007), [Cu(bipy)][V_2O_6] and [Cu(bipy)$_2$][V_2O_6] (DeBord *et al.*, 1996), [Mn(phen)$_2$]$_2$[V_4O_{12}].0.5H_2O (Lu *et al.*, 2002), and [Co(phen)$_2$]$_2$[V_4O_{12}].H_2O (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)]$_2$$V_4O_{12}$}$_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 V_4O_{12} unit (Fig. 1). The V_4O_{12} units are linked through six square-pyramidal Cu^{II} atoms to six adjacent V_4O_{12} rings (Fig. 2). Two of VO_4 units in the V_4O_{12} unit each connect with one square-pyramidal Cu unit, while the other two VO_4 units each exhibit corner-sharing interactions with two Cu units. Each Cu unit links three V_4O_{12} 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 angel 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 V_2O_5 (0.73 g, 4.0 mmol), 2,2'-bipyridine dihydrate (0.38 g, 2.0 mmol), $CuCl_2$.2H_2O (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 = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

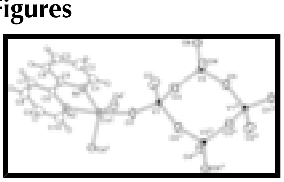

Fig. 1. The asymmetric unit of the title compound, extended to show the V_4O_{12} unit. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) *x*, *y* - 1, *z*; (iii) 2 - *x*, 1 - *y*, 1 - *z*.]

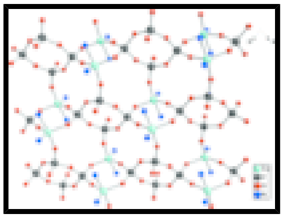

Fig. 2. A view of the copper–vanadate layer with C and H atoms of the bipy ligands omitted for clarity.

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

Crystal data

$[Cu_2V_4O_{12}(C_{10}H_8N_2)_2]$	$Z = 2$
$M_r = 417.60$	$F_{000} = 410$
Triclinic, $P\overline{1}$	$D_x = 2.137$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.1019$ (4) Å	Cell parameters from 3811 reflections
$b = 8.3122$ (5) Å	$\theta = 2.1$–28.3°
$c = 10.3501$ (4) Å	$\mu = 3.06$ mm^{-1}
$\alpha = 72.332$ (3)°	$T = 298$ (2) K
$\beta = 84.562$ (3)°	Block, green
$\gamma = 77.878$ (3)°	0.33 × 0.31 × 0.25 mm
$V = 648.98$ (6) Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	3114 independent reflections
Radiation source: fine-focus sealed tube	2553 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.055$
$T = 298(2)$ K	$\theta_{max} = 28.3$°
φ and ω scans	$\theta_{min} = 2.1$°
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -10 \rightarrow 9$
$T_{min} = 0.379$, $T_{max} = 0.469$	$k = -10 \rightarrow 7$
4603 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.084$	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
3114 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}

190 parameters	$\Delta\rho_{min}$ = −0.75 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å^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)

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 (Å, °)

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)

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

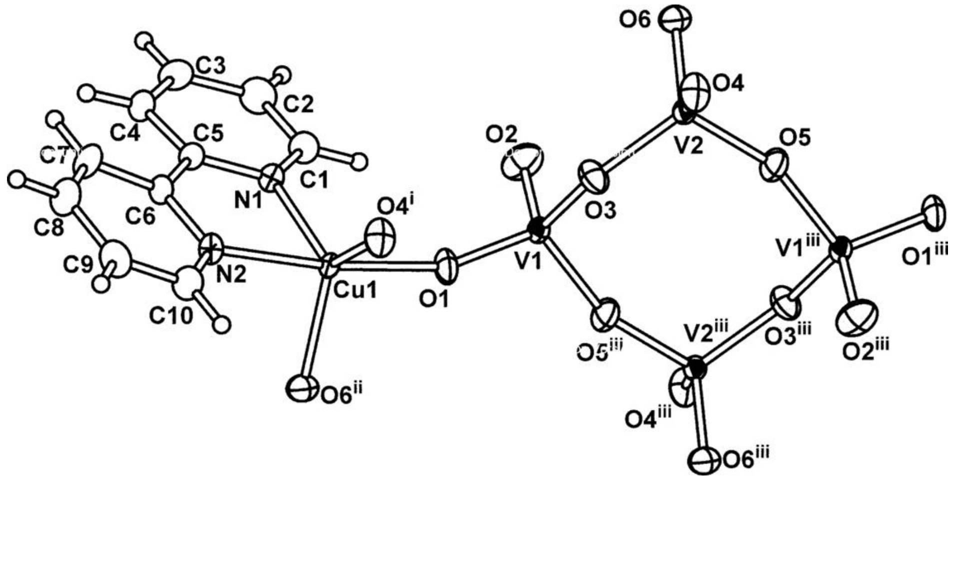
O6
O4
C3
C2
C4
C5
V2
O5
O2
C7
C1
N1
O4i
C8
C6
O3
V1
V1iii
O1iii
C9
N2
Cu1
O1
C10
O5iii
V2iii
O3iii
O2iii
O6ii
O4iii
O6iii

Fig. 2