

catena-Poly[[[(2,2'-bipyridyl)copper(II)]- μ -L-alaninato] perchlorate monohydrate]Mircea Braban,^{a*} Ionel Haiduc^a and Peter Lönnecke^b

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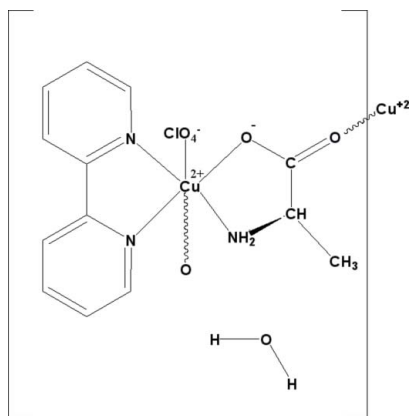
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Key indicators: single-crystal X-ray study; $T = 220$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.033; wR factor = 0.078; data-to-parameter ratio = 13.6.

In the structure of the polymeric title complex, $[\text{Cu}(\text{C}_3\text{H}_6\text{NO}_2)(\text{C}_{10}\text{H}_8\text{N}_2)]\text{ClO}_4 \cdot \text{H}_2\text{O}$, the carboxylate group of the chelating amino acid is further linked to a neighbouring Cu centre, generating a supramolecular single-stranded chain parallel to [010]. The structure displays intermolecular N—H...O and O—H...O hydrogen bonding, which consolidates the crystal packing.

Related literature

For related structures, see: Antolini *et al.* (1983); Masuda *et al.* (1991); Sgarabotto *et al.* (1999); Solans *et al.* (1992).

**Experimental***Crystal data*

$[\text{Cu}(\text{C}_3\text{H}_6\text{NO}_2)(\text{C}_{10}\text{H}_8\text{N}_2)]\text{ClO}_4 \cdot \text{H}_2\text{O}$
 $M_r = 425.28$

Monoclinic, $P2_1/c$
 $a = 13.1807$ (10) Å
 $b = 8.2656$ (6) Å

$c = 16.1195$ (13) Å
 $\beta = 110.606$ (2)°
 $V = 1643.8$ (2) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 1.53$ mm⁻¹
 $T = 220$ (2) K
 $0.60 \times 0.30 \times 0.30$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.460$, $T_{\max} = 0.656$

13671 measured reflections
3939 independent reflections
3611 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.078$
 $S = 1.11$
3939 reflections

290 parameters
All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.89$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H2N3}\cdots\text{O7}^i$	0.74 (4)	2.60 (4)	3.293 (4)	159 (5)
$\text{N3}-\text{H1N3}\cdots\text{O1}^{ii}$	0.83 (4)	2.48 (4)	3.225 (3)	149 (3)
$\text{N3}-\text{H1N3}\cdots\text{O2}^{ii}$	0.83 (4)	2.91 (4)	3.225 (3)	105 (3)
$\text{N3}-\text{H1N3}\cdots\text{O7}^{ii}$	0.83 (4)	2.70 (5)	3.059 (3)	108 (3)
$\text{N3}-\text{H2N3}\cdots\text{O7}^{ii}$	0.74 (4)	2.69 (5)	3.059 (3)	114 (4)
$\text{O7}-\text{H1O7}\cdots\text{O2}^{ii}$	0.79 (4)	2.08 (4)	2.857 (3)	166 (3)

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and DIAMOND (Brandenburg, 1999); software used to prepare material for publication: WinGX (Farrugia, 1999).

The authors thank Professor Evamarie Hey-Hawkins for cooperation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2300).

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supplementary materials

Acta Cryst. (2009). E65, m51 [doi:10.1107/S1600536808040725]

***catena*-Poly[[[(2,2'-bipyridyl)copper(II)]- μ -L-alaninato] perchlorate monohydrate]**

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Comment

The structure of the title complex, (I) and Fig. 1, is of interest with respect to the stereochemistry of the complexed aminoacid, the coordination geometry of the metal centre and the single-stranded supramolecular assembly created primarily by further coordination of the carboxylate group of the aminoacid, Fig. 2. The secondary association is by crosslinks realised through H-bonds between the created chains, Fig. 3. The supramolecular structure described for (I) is found in other (aminoacidato)(2,2'-bipyridyl)copper(II) complexes, such as in the tryptophanato (Masuda *et al.*, 1991) and aspartato complexes (Antolini *et al.*, 1983). The assembly has also been identified in the proline complex but not described as a supramolecular association (Sgarabotto *et al.*, 1999). For the alaninate complex, see also Solans *et al.* (1992).

Experimental

The synthesis of (I) was realized by using an intermediate complex, i.e. tris(2,2'-bipyridyl)copper(II), as shown in Fig. 4. The cation in (I) was prepared according to the following procedure: Two ethanolic solutions, one containing 2,2'-bipyridyl (0.31 g, 2 mmol/5 mL) and another containing $\text{Cu}(\text{ClO}_4)_2 \cdot 2\text{H}_2\text{O}$ (0.6 g, 2 mmol/5 mL) were mixed with stirring. To the resulting suspension of a blue powder, an alkaline solution of L-alanine (0.18 g alanine + 0.08 g NaOH 2 mmol/10 mL water) was added dropwise (see also Scheme 2). The suspension cleared and changed colour to dark-blue. The mixture was heated to 50°C and Na_2ClO_4 (1 mmol) was added. After 10 mins, the solution was cooled and filtered. The filtrate was allowed to stand at room temperature for several days when dark-blue crystals, suitable for X-ray analysis, separated, collected and washed with a methanolic solution.

Refinement

The H atoms were refined freely: O-H = 0.69 (5) - 0.79 (3) Å, N-H = 0.73 (4) - 0.83 (3) Å, and C-H = 0.89 (3) - 1.16 (4) Å.

Figures

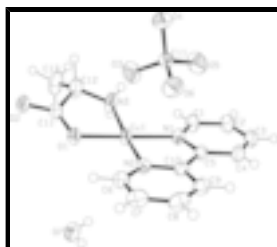


Fig. 1. The asymmetric unit in (I) showing the crystallographic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

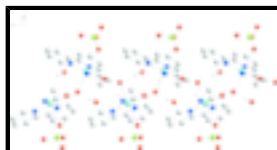


Fig. 2. Single strand supramolecular assembly mediated by further coordination of the carboxylate group of the aminoacid. Colour code: cyan = Cu, green = Cl, red = O, blue = N, grey = C. Hydrogen atoms have been omitted for clarity.

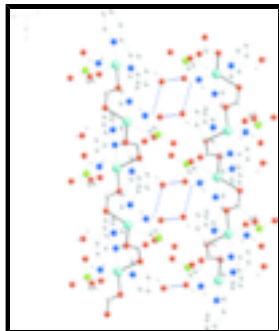


Fig. 3. Supramolecular assembly at the secondary level formed by H-bond formation. The single strand chain is represented with thick bonds whereas the H-bonds are represented by blue dashed lines. For clarity only hydrogens (shown in white) involved in intermolecular associations are represented.

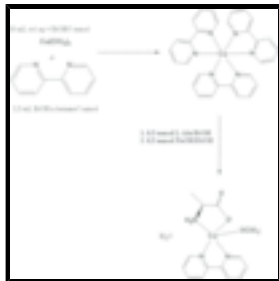


Fig. 4. The formation of the title compound.

catena-Poly[[[(2,2'-bipyridyl)copper(II)]- μ -L-alaninato] perchlorate monohydrate]

Crystal data

[Cu(C₃H₆NO₂)(C₁₀H₈N₂)]ClO₄·H₂O

M_r = 425.28

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

a = 13.1807 (10) Å

b = 8.2656 (6) Å

c = 16.1195 (13) Å

β = 110.606 (2)°

V = 1643.8 (2) Å³

Z = 4

F_{000} = 868

D_x = 1.718 Mg m⁻³

Melting point: 253 K

Mo $K\alpha$ radiation

λ = 0.71073 Å

Cell parameters from 4860 reflections

θ = 2.7–28.3°

μ = 1.53 mm⁻¹

T = 220 (2) K

Prism, dark blue

0.60 × 0.30 × 0.30 mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 81.92 pixels mm⁻¹

T = 220(2) K

φ scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1997)

T_{\min} = 0.460, T_{\max} = 0.656

13671 measured reflections

3939 independent reflections

3611 reflections with $I > 2\sigma(I)$

R_{int} = 0.020

θ_{max} = 28.3°

θ_{min} = 2.6°

h = -17→17

k = -11→11

l = -20→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.033$	All H-atom parameters refined
$wR(F^2) = 0.078$	$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 1.4344P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
3939 reflections	$(\Delta/\sigma)_{\max} = 0.001$
290 parameters	$\Delta\rho_{\max} = 0.89 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$
	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.364703 (18)	0.28840 (3)	0.167573 (16)	0.02538 (8)
Cl1	0.17905 (4)	0.19812 (6)	-0.06197 (3)	0.02952 (11)
O1	0.43017 (11)	0.07522 (17)	0.20438 (10)	0.0300 (3)
O2	0.55154 (13)	-0.09670 (18)	0.18782 (10)	0.0365 (3)
O3	0.28355 (13)	0.1556 (3)	0.00253 (12)	0.0505 (4)
O4	0.16505 (15)	0.1197 (2)	-0.14439 (11)	0.0450 (4)
O5	0.17426 (19)	0.3705 (2)	-0.07423 (13)	0.0605 (5)
O6	0.09645 (14)	0.1466 (3)	-0.02883 (12)	0.0510 (4)
N1	0.28664 (13)	0.4972 (2)	0.12453 (11)	0.0275 (3)
N2	0.22426 (13)	0.2334 (2)	0.18192 (11)	0.0259 (3)
N3	0.49264 (17)	0.3247 (2)	0.13184 (19)	0.0391 (5)
C1	0.32627 (18)	0.6292 (3)	0.09841 (16)	0.0358 (5)
C2	0.2644 (2)	0.7669 (3)	0.06765 (17)	0.0398 (5)
C3	0.1579 (2)	0.7682 (3)	0.06305 (16)	0.0382 (5)
C4	0.11629 (18)	0.6331 (3)	0.09049 (15)	0.0350 (5)
C5	0.18218 (15)	0.4985 (2)	0.12098 (12)	0.0264 (4)
C6	0.19879 (18)	0.0907 (3)	0.20930 (14)	0.0330 (4)
C7	0.0946 (2)	0.0558 (3)	0.20633 (16)	0.0391 (5)
C8	0.01505 (19)	0.1715 (3)	0.17510 (16)	0.0391 (5)

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C9	0.04044 (17)	0.3198 (3)	0.14800 (14)	0.0340 (4)
C10	0.14634 (15)	0.3474 (2)	0.15143 (12)	0.0264 (4)
C11	0.50804 (16)	0.0390 (2)	0.17798 (13)	0.0288 (4)
C12	0.5466 (2)	0.1693 (3)	0.12787 (19)	0.0429 (5)
C13	0.6666 (2)	0.1814 (4)	0.1552 (3)	0.0605 (8)
H2N3	0.482 (4)	0.357 (6)	0.087 (3)	0.098 (16)*
H1N3	0.536 (3)	0.384 (5)	0.170 (3)	0.083 (13)*
H1	0.398 (2)	0.628 (3)	0.1013 (17)	0.042 (7)*
H2	0.293 (2)	0.844 (4)	0.0488 (19)	0.048 (8)*
H3	0.116 (2)	0.855 (4)	0.0419 (17)	0.044 (7)*
H4	0.049 (2)	0.634 (4)	0.0873 (19)	0.054 (8)*
H6	0.257 (2)	0.014 (3)	0.2313 (16)	0.037 (6)*
H7	0.082 (2)	-0.043 (4)	0.2244 (18)	0.045 (7)*
H8	-0.057 (2)	0.151 (4)	0.1713 (18)	0.052 (8)*
H9	-0.014 (2)	0.401 (4)	0.1242 (18)	0.046 (7)*
H12	0.539 (3)	0.124 (5)	0.059 (3)	0.094 (12)*
H13A	0.691 (3)	0.261 (4)	0.116 (2)	0.073 (10)*
H13B	0.699 (3)	0.072 (5)	0.145 (2)	0.077 (11)*
H13C	0.691 (4)	0.225 (5)	0.228 (3)	0.105 (15)*
O7	0.4174 (2)	0.1391 (3)	0.41504 (15)	0.0516 (5)
H1O7	0.418 (3)	0.220 (4)	0.389 (2)	0.056 (10)*
H2O7	0.367 (4)	0.142 (7)	0.419 (3)	0.110 (19)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02196 (12)	0.01912 (12)	0.03468 (14)	0.00188 (8)	0.00948 (9)	0.00357 (9)
Cl1	0.0286 (2)	0.0299 (2)	0.0284 (2)	0.00289 (17)	0.00796 (18)	0.00106 (18)
O1	0.0300 (7)	0.0218 (7)	0.0391 (8)	0.0031 (5)	0.0133 (6)	0.0050 (6)
O2	0.0399 (8)	0.0251 (7)	0.0431 (8)	0.0101 (6)	0.0128 (7)	0.0037 (6)
O3	0.0284 (8)	0.0680 (13)	0.0458 (9)	0.0050 (8)	0.0016 (7)	0.0056 (9)
O4	0.0561 (10)	0.0443 (10)	0.0355 (8)	0.0040 (8)	0.0171 (7)	-0.0061 (7)
O5	0.0954 (16)	0.0274 (9)	0.0515 (11)	0.0022 (9)	0.0167 (10)	0.0023 (8)
O6	0.0375 (9)	0.0659 (13)	0.0562 (11)	0.0028 (8)	0.0248 (8)	0.0028 (9)
N1	0.0251 (8)	0.0227 (8)	0.0333 (8)	0.0019 (6)	0.0082 (6)	0.0011 (6)
N2	0.0253 (8)	0.0245 (8)	0.0276 (8)	-0.0015 (6)	0.0088 (6)	-0.0017 (6)
N3	0.0310 (10)	0.0249 (9)	0.0666 (15)	0.0059 (7)	0.0237 (10)	0.0115 (10)
C1	0.0320 (11)	0.0268 (10)	0.0480 (12)	0.0003 (8)	0.0132 (9)	0.0051 (9)
C2	0.0467 (13)	0.0245 (10)	0.0470 (13)	0.0005 (9)	0.0149 (10)	0.0051 (9)
C3	0.0416 (12)	0.0288 (11)	0.0378 (11)	0.0118 (9)	0.0062 (9)	0.0029 (9)
C4	0.0287 (10)	0.0347 (11)	0.0372 (11)	0.0091 (9)	0.0060 (8)	-0.0004 (9)
C5	0.0245 (9)	0.0263 (9)	0.0257 (9)	0.0023 (7)	0.0054 (7)	-0.0027 (7)
C6	0.0356 (11)	0.0284 (10)	0.0353 (10)	-0.0028 (8)	0.0131 (9)	-0.0010 (8)
C7	0.0436 (12)	0.0347 (12)	0.0442 (12)	-0.0134 (10)	0.0220 (10)	-0.0047 (10)
C8	0.0299 (10)	0.0483 (13)	0.0425 (12)	-0.0106 (10)	0.0168 (9)	-0.0093 (10)
C9	0.0254 (9)	0.0426 (12)	0.0337 (10)	-0.0007 (9)	0.0098 (8)	-0.0057 (9)
C10	0.0248 (9)	0.0289 (10)	0.0245 (8)	-0.0008 (7)	0.0074 (7)	-0.0049 (7)
C11	0.0261 (9)	0.0247 (9)	0.0309 (9)	0.0011 (7)	0.0042 (7)	0.0014 (8)

C12	0.0388 (12)	0.0332 (12)	0.0623 (15)	0.0089 (9)	0.0249 (11)	0.0115 (11)
C13	0.0427 (14)	0.0452 (15)	0.105 (3)	0.0125 (12)	0.0408 (16)	0.0190 (16)
O7	0.0602 (13)	0.0449 (11)	0.0585 (12)	0.0192 (9)	0.0318 (10)	0.0107 (9)

Geometric parameters (Å, °)

Cu1—O1	1.9598 (14)	C2—H2	0.85 (3)
Cu1—N3	1.987 (2)	C3—C4	1.384 (4)
Cu1—N2	1.9970 (16)	C3—H3	0.90 (3)
Cu1—N1	2.0043 (17)	C4—C5	1.390 (3)
Cu1—O2 ⁱ	2.3965 (16)	C4—H4	0.86 (3)
Cl1—O4	1.4307 (16)	C5—C10	1.479 (3)
Cl1—O6	1.4358 (18)	C6—C7	1.387 (3)
Cl1—O5	1.4365 (19)	C6—H6	0.96 (3)
Cl1—O3	1.4469 (16)	C7—C8	1.377 (4)
O1—C11	1.277 (2)	C7—H7	0.90 (3)
O2—C11	1.244 (2)	C8—C9	1.381 (3)
O2—Cu1 ⁱⁱ	2.3965 (16)	C8—H8	0.95 (3)
N1—C1	1.340 (3)	C9—C10	1.396 (3)
N1—C5	1.358 (2)	C9—H9	0.96 (3)
N2—C6	1.343 (3)	C11—C12	1.536 (3)
N2—C10	1.353 (3)	C12—C13	1.488 (4)
N3—C12	1.481 (3)	C12—H12	1.15 (4)
N3—H2N3	0.74 (4)	C13—H13A	1.04 (4)
N3—H1N3	0.83 (4)	C13—H13B	1.04 (4)
C1—C2	1.386 (3)	C13—H13C	1.16 (5)
C1—H1	0.92 (3)	O7—H107	0.79 (4)
C2—C3	1.380 (4)	O7—H207	0.69 (5)
O1—Cu1—N3	83.99 (7)	C4—C3—H3	120.4 (18)
O1—Cu1—N2	95.03 (6)	C3—C4—C5	119.4 (2)
N3—Cu1—N2	169.62 (10)	C3—C4—H4	119 (2)
O1—Cu1—N1	175.41 (6)	C5—C4—H4	122 (2)
N3—Cu1—N1	98.87 (7)	N1—C5—C4	121.28 (19)
N2—Cu1—N1	81.51 (7)	N1—C5—C10	114.65 (16)
O1—Cu1—O2 ⁱ	93.37 (6)	C4—C5—C10	124.07 (19)
N3—Cu1—O2 ⁱ	94.28 (9)	N2—C6—C7	122.0 (2)
N2—Cu1—O2 ⁱ	96.09 (6)	N2—C6—H6	116.2 (16)
N1—Cu1—O2 ⁱ	90.00 (6)	C7—C6—H6	121.8 (16)
O4—Cl1—O6	110.13 (11)	C8—C7—C6	119.0 (2)
O4—Cl1—O5	109.66 (11)	C8—C7—H7	123.1 (18)
O6—Cl1—O5	110.07 (13)	C6—C7—H7	117.9 (18)
O4—Cl1—O3	109.65 (11)	C7—C8—C9	119.6 (2)
O6—Cl1—O3	108.38 (11)	C7—C8—H8	121.0 (19)
O5—Cl1—O3	108.93 (13)	C9—C8—H8	119.4 (19)
C11—O1—Cu1	115.46 (12)	C8—C9—C10	118.9 (2)
C11—O2—Cu1 ⁱⁱ	121.01 (14)	C8—C9—H9	120.8 (17)
C1—N1—C5	118.82 (17)	C10—C9—H9	120.3 (17)

supplementary materials

C1—N1—Cu1	126.92 (14)	N2—C10—C9	121.42 (19)
C5—N1—Cu1	114.25 (13)	N2—C10—C5	114.75 (16)
C6—N2—C10	119.06 (18)	C9—C10—C5	123.82 (19)
C6—N2—Cu1	125.95 (14)	O2—C11—O1	123.87 (19)
C10—N2—Cu1	114.56 (13)	O2—C11—C12	118.42 (19)
C12—N3—Cu1	110.57 (14)	O1—C11—C12	117.66 (18)
C12—N3—H2N3	101 (4)	N3—C12—C13	113.9 (2)
Cu1—N3—H2N3	117 (3)	N3—C12—C11	109.41 (19)
C12—N3—H1N3	109 (3)	C13—C12—C11	113.9 (2)
Cu1—N3—H1N3	108 (3)	N3—C12—H12	116 (2)
H2N3—N3—H1N3	111 (4)	C13—C12—H12	91.9 (19)
N1—C1—C2	122.4 (2)	C11—C12—H12	111 (2)
N1—C1—H1	118.5 (18)	C12—C13—H13A	113 (2)
C2—C1—H1	119.1 (18)	C12—C13—H13B	111 (2)
C3—C2—C1	119.0 (2)	H13A—C13—H13B	102 (3)
C3—C2—H2	123 (2)	C12—C13—H13C	102 (2)
C1—C2—H2	118 (2)	H13A—C13—H13C	113 (3)
C2—C3—C4	119.1 (2)	H13B—C13—H13C	117 (3)
C2—C3—H3	120.5 (18)	H1O7—O7—H2O7	102 (5)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H2N3 \cdots O7 ⁱⁱⁱ	0.74 (4)	2.60 (4)	3.293 (4)	159 (5)
N3—H1N3 \cdots O1 ⁱ	0.83 (4)	2.48 (4)	3.225 (3)	149 (3)
N3—H1N3 \cdots O2 ⁱ	0.83 (4)	2.91 (4)	3.225 (3)	105 (3)
N3—H1N3 \cdots O7 ⁱ	0.83 (4)	2.70 (5)	3.059 (3)	108 (3)
N3—H2N3 \cdots O7 ⁱ	0.74 (4)	2.69 (5)	3.059 (3)	114 (4)
O7—H1O7 \cdots O2 ⁱ	0.79 (4)	2.08 (4)	2.857 (3)	166 (3)

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

Fig. 1

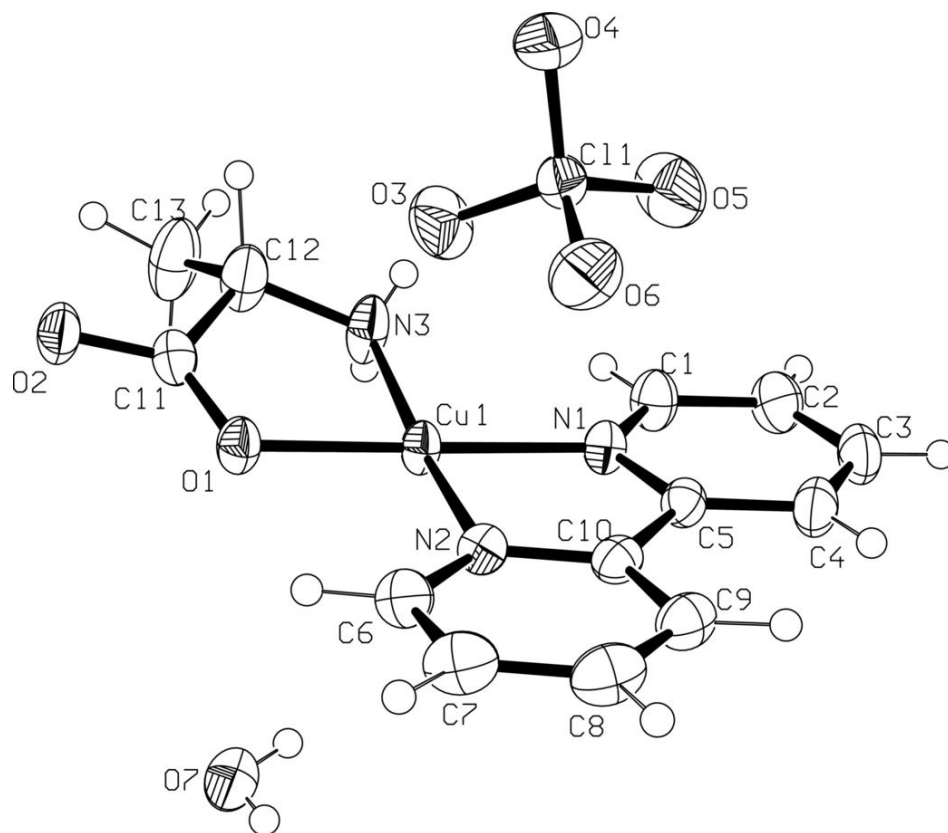


Fig. 2

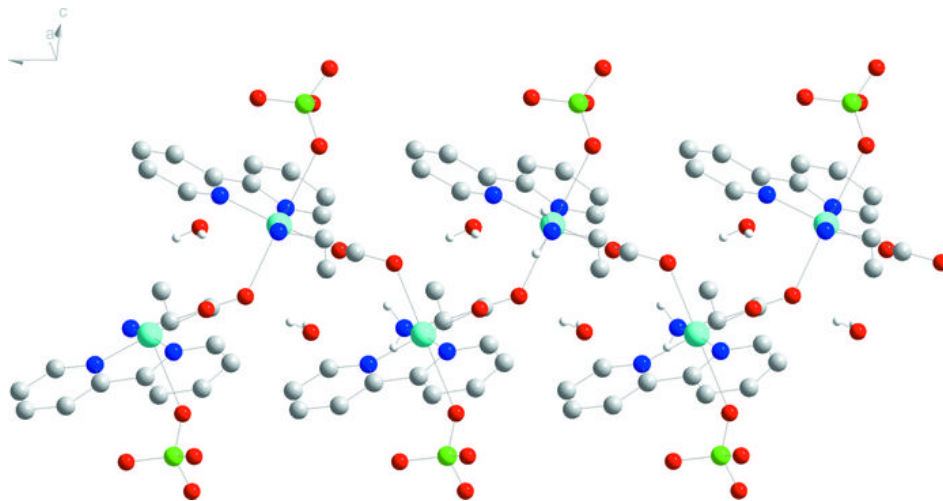


Fig. 3

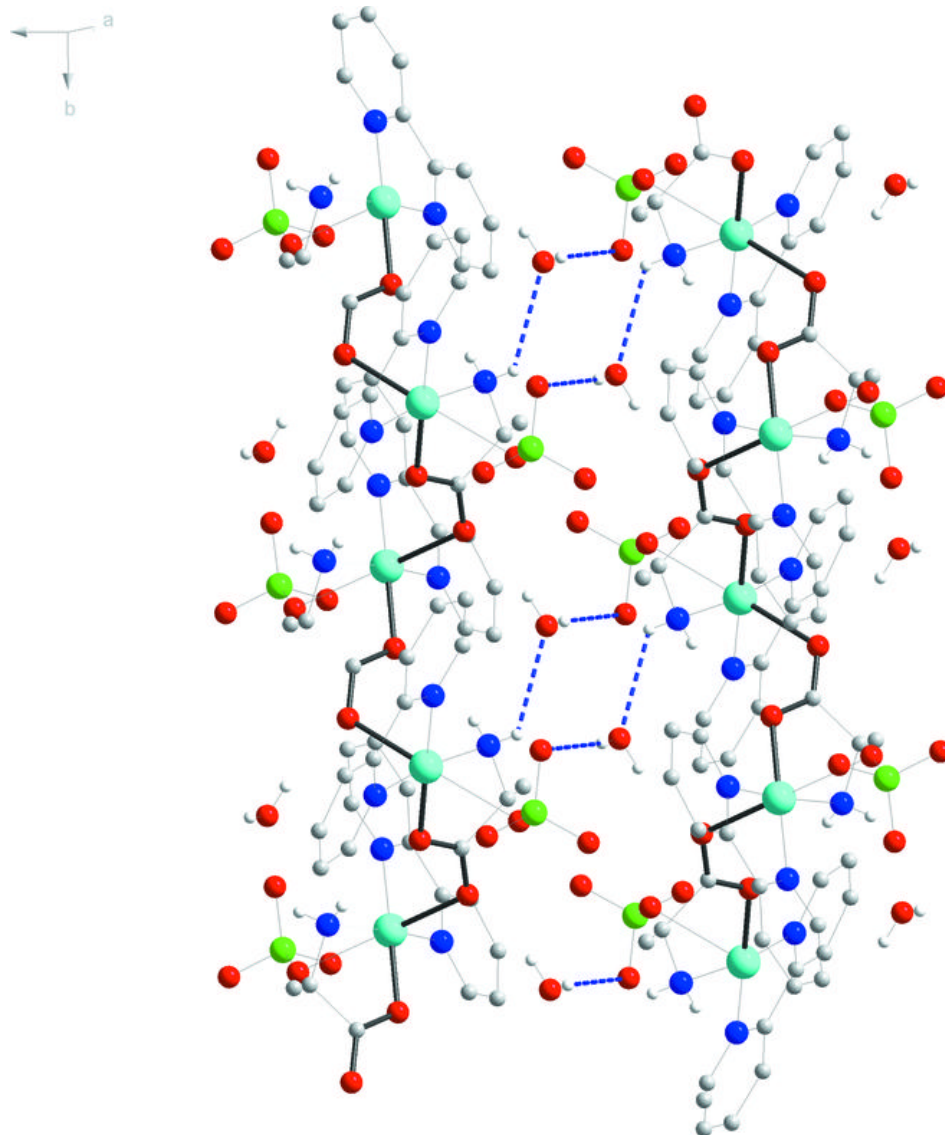


Fig. 4

