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Aqua[tris(1*H*-benzimidazol-2-ylmethyl)-amine]copper(II) diperchlorate 4-picoline *N*-oxide monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.054
wR factor = 0.171
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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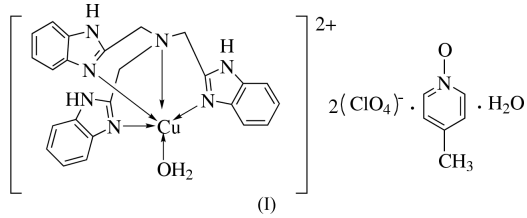
In the title compound, $[Cu(C_{24}H_{21}N_7)(H_2O)](ClO_4)_2\cdot C_6H_7NO\cdot H_2O$, the copper ion is bonded to a tris(1*H*-benzimidazol-2-ylmethyl)amine (ntb) and a water molecule, resulting in it being five-coordinate with an N_4O ligand set. The coordination geometry of the copper ion may best be described as distorted trigonal bipyramidal, with *C*3 molecular symmetry.

Comment

The asymmetric unit of the title compound, (I) (Fig. 1), consists of a discrete $[Cu(ntb)(H_2O)]^{2+}$ cation [ntb is tris(1*H*-benzimidazol-2-ylmethyl)amine], two perchlorate anions, a molecule of 4-picoline N-oxide and one molecule of water of crystallization. The water molecule is disordered over two sites with equal occupancies. The copper ion is five-coordinate with an N_4O ligand set. The ntb ligand acts as a tetradentate N-atom donor, and an aqua O atom completes the coordination. The coordination geometry of the copper may best be described as distorted trigonal bipyramidal, with approximate site symmetry *C*3. This geometry is assumed by the copper center to relieve the steric crowding. The trigonal plane is occupied by the three ligating N atoms of the benzimidazolyl groups. The Cu atom protrudes towards atom O1 and is 0.294 Å out of the plane. The axial ligating atoms are N1 and O1, with Cu—N1 = 2.096 (3) Å, Cu—O1 = 1.959 (3) Å and N1—Cu1—O1 = 178.71 (11)°. The three benzimidazole ring arms of the ntb ligand form a cone-shaped cavity. The bond lengths and angles are normal. In the crystal structure, there are weak C—H···O and strong N—H···O and O—H···O hydrogen bonds (Table 1 and Fig. 2).



(I)

Experimental

To a stirred solution of tris(1*H*-benzimidazol-2-ylmethyl)amine (407 mg, 1 mmol) in methanol (20 ml) was added $Cu(ClO_4)_2\cdot 6H_2O$ (370 mg, 1 mmol), followed by a solution of 4-picoline *N*-oxide (109 mg, 1 mmol) in methanol (5 ml). The resulting clear blue solution was stirred for 8 h and then allowed to stand at room temperature. Blue–green crystals suitable for X-ray diffraction studies were obtained after two weeks (367 mg, yield 45%). Found: C 43.82, H 3.99, N 13.93%; calculated for $C_{30}H_{32}Cl_2CuN_8O_{11}$: C 44.21, H 3.96, N 13.75%.

Crystal data

$[Cu(C_{24}H_{21}N_7)(H_2O)]$-$(ClO_4)_2\cdot C_6H_7NO\cdot H_2O$	$Z = 2$
$M_r = 815.08$	$D_x = 1.485$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 11.6048$ (13) Å	Cell parameters from 2688 reflections
$b = 13.8916$ (15) Å	$\theta = 2.3$–$23.1°$
$c = 14.3273$ (16) Å	$\mu = 0.81$ mm^{-1}
$\alpha = 62.430$ (2)°	$T = 293$ (2) K
$\beta = 89.898$ (2)°	Cuboid, blue–green
$\gamma = 66.140$ (2)°	0.3 × 0.2 × 0.2 mm
$V = 1822.8$ (4) Å^3	

Data collection

Bruker SMART CCD diffractometer	6971 independent reflections
φ and ω scans	4936 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$R_{int} = 0.026$
$T_{min} = 0.82$, $T_{max} = 0.85$	$\theta_{max} = 26.0°$
9837 measured reflections	$h = -14 \rightarrow 13$
	$k = -17 \rightarrow 11$
	$l = -17 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1057P)^2 + 0.3685P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.054$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.171$	$\Delta\rho_{max} = 0.31$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
6971 reflections	
485 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1W	0.86	2.01	2.844 (6)	163
N4—H4A···O2	0.86	1.96	2.728 (4)	147
N4—H4A···O23^{i}	0.86	2.48	2.935 (4)	114
N6—H6A···O13	0.86	2.16	3.015 (4)	170
N6—H6A···O12	0.86	2.49	3.148 (4)	134
O1—H1C···O2^{ii}	0.81 (3)	1.81 (3)	2.620 (4)	177 (5)
O1—H1D···O12^{iii}	0.81 (3)	2.07 (3)	2.852 (4)	162 (4)
O1W—H1WB···O21	0.85	1.98	2.830 (6)	180
O1W—H1WA···O2W^{iv}	0.85	2.48	3.288 (8)	158
C14—H14···O11^{v}	0.93	2.52	3.259 (5)	136

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

H atoms bonded to the coordinated water molecule were included in the refinement with both O—H distances restrained to be equal. All other H atoms were placed in calculated positions, with C—H distances ranging from 0.93 to 1.00 Å, N—H = 0.86 Å and O—H = 0.85 Å, and treated as riding, with $U_{iso} = 1.2U_{eq}$ ($1.5U_{eq}$ for methyl H atoms) of the carrier atom. .

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2004) and *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

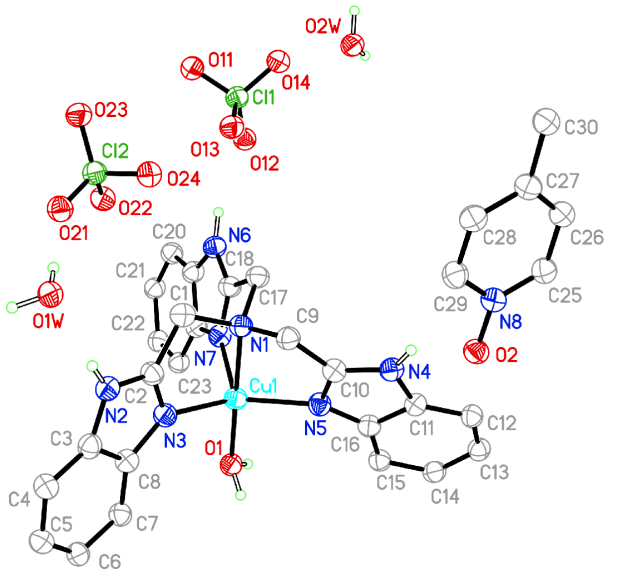


Figure 1
View of the asymmetric unit of the title compound. Displacement ellipsoids are at the 30% probability level and H atoms bonded to C atoms are not shown

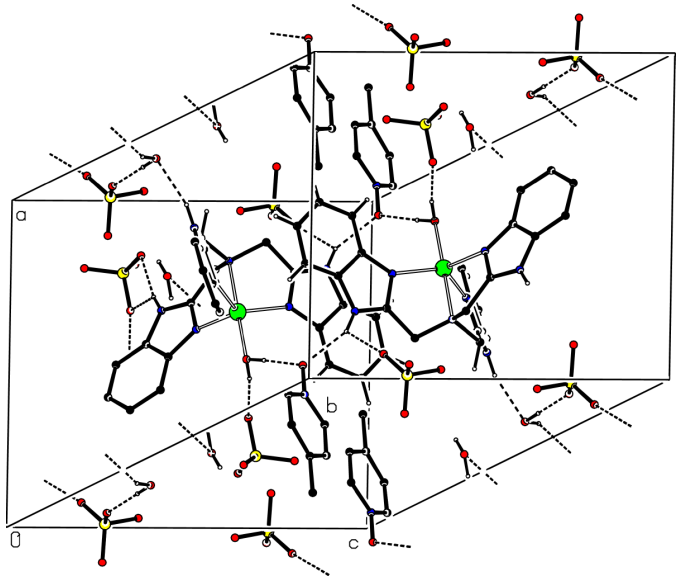


Figure 2
Packing diagram (Spek, 2004) of the title crystal structure, with hydrogen bonds shown as dashed lines. Colour codes: green Cu, yellow Cl, red O, blue N and black C.

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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