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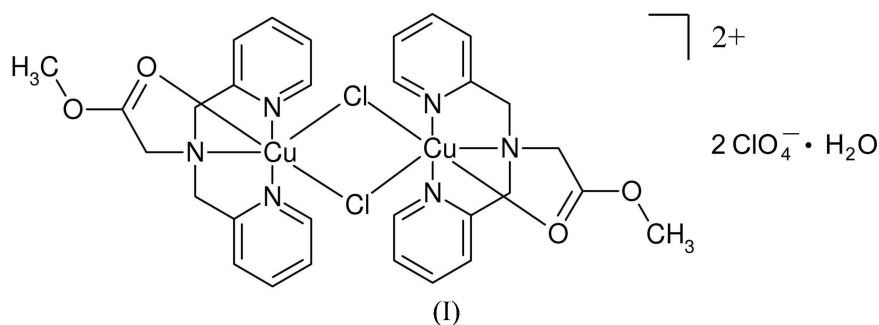
Key indicators

Single-crystal X-ray study
 $T = 180$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.048
 wR factor = 0.116
Data-to-parameter ratio = 17.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Di- μ -chloro-bis({methyl [N,N -bis(2-pyridyl-
methyl)amino]acetate- $\kappa^4 N,N',N'',O$ })copper(II))
bis(perchlorate) monohydrateThe title compound, $[\text{Cu}_2\text{Cl}_2(\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_2)_2](\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$, contains dichloro-bridged Cu^{II} complexes located on centres of inversion. The solvent water molecule lies on a twofold rotation axis and forms hydrogen bonds to two perchlorate anions.

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Comment

In our previous report (Nielsen *et al.*, 2006), we described the dichloro-bridged Cu^{II} complex $[\text{Cu}_2\text{Cl}_2(\text{bpgH})_2](\text{ClO}_4)_2 \cdot 4\text{CH}_3\text{CN}$, where bpgH is the tetradentate bis(2-pyridylmethyl)glycine ligand. The title compound, (I), is closely comparable, but with esterification of the carboxylate groups. Compound (I) contains dichloro-bridged dicopper(II) complexes (Fig. 1), lying on centres of inversion. The coordination geometry around Cu^{II} (Table 1) is closely comparable to that of the bpgH complex (Nielsen *et al.*, 2006), being approximately octahedral with significant elongation of the $\text{Cu1}-\text{O1}$ and $\text{Cu1}-\text{Cl1}^i$ bonds [symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, -z$] on account of the Jahn–Teller distortion associated with Cu^{II} . The uncoordinated water molecule lies on a twofold rotation axis and forms hydrogen bonds to two perchlorate anions (Table 2).

Experimental

Esterification of the bpgH ligand to give methyl [bis(2-pyridylmethyl)amino]acetate (bpe) was achieved by a procedure similar to that reported by Rodriguez *et al.* (1997). $\text{Fe}(\text{ClO}_4)_3 \cdot 9\text{H}_2\text{O}$ (0.1103 g, 0.214 mmol) in 0.5 ml acetonitrile was added to bpgH {bpgH = [bis(2-pyridylmethyl)amino]acetate, 0.1099 g, 0.213 mmol} in 2.5 ml hot acetonitrile. The initial orange precipitate was dissolved by addition of 1 ml water and one drop of 70% HClO_4 . The solvent volume was reduced by evaporation under gentle heating and a fine orange precipitate of $[\text{Fe}(\text{bpe})\text{Cl}_3]$ was filtered off (yield 0.021 g). The metathesis reaction (replacing Fe by Cu) was carried out by dissolving the orange solid in a mixture of hot methanol (2.5 ml) and water (0.5 ml), and adding $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.0078 g, 0.046 mmol) in 0.25 ml methanol. Turquoise crystals were formed after 1–2 d [total yield of

(I) 0.0104 g; ca 10% based on bpgH]. IR (KBr, cm^{-1}): $\nu(\text{C}=\text{O})$ 1612 (s).

Crystal data

$[\text{Cu}_2\text{Cl}_2(\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_2)_2] \cdot (\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$
 $M_r = 957.53$
 Monoclinic, $C2/c$
 $a = 19.1398$ (7) Å
 $b = 8.5993$ (3) Å
 $c = 24.1605$ (8) Å
 $\beta = 111.380$ (1)°

$V = 3702.9$ (2) Å³
 $Z = 4$
 $D_x = 1.718$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 1.51$ mm⁻¹
 $T = 180$ (2) K
 Block, turquoise
 0.25 × 0.08 × 0.08 mm

Data collection

Bruker–Nonius X8-APEX-II CCD diffractometer
 Thin-slice ω and φ scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\text{min}} = 0.735$, $T_{\text{max}} = 0.889$

36620 measured reflections
 4535 independent reflections
 3461 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.116$
 $S = 1.11$
 4535 reflections
 254 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 16.3749P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cu1–N1	1.992 (3)	Cu1–O1	2.386 (3)
Cu1–N2	2.068 (3)	Cu1–Cl1	2.2564 (8)
Cu1–N3	1.999 (3)	Cu1–Cl1 ⁱ	2.9424 (9)
N1–Cu1–N2	83.32 (12)	N3–Cu1–O1	94.26 (10)
N1–Cu1–N3	164.41 (11)	Cl1–Cu1–O1	100.28 (7)
N2–Cu1–N3	81.76 (12)	N1–Cu1–Cl1 ⁱ	86.91 (8)
N1–Cu1–Cl1	98.23 (8)	N2–Cu1–Cl1 ⁱ	90.04 (8)
N2–Cu1–Cl1	178.45 (9)	N3–Cu1–Cl1 ⁱ	88.60 (8)
N3–Cu1–Cl1	96.70 (8)	Cl1–Cu1–Cl1 ⁱ	90.00 (3)
N1–Cu1–O1	87.51 (11)	O1–Cu1–Cl1 ⁱ	168.91 (7)
N2–Cu1–O1	79.80 (10)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1W} \cdots \text{H1W} \cdots \text{O2A}$	0.84 (6)	2.18 (2)	2.951 (7)	153 (2)

H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with $\text{C}-\text{H} = 0.95$ Å

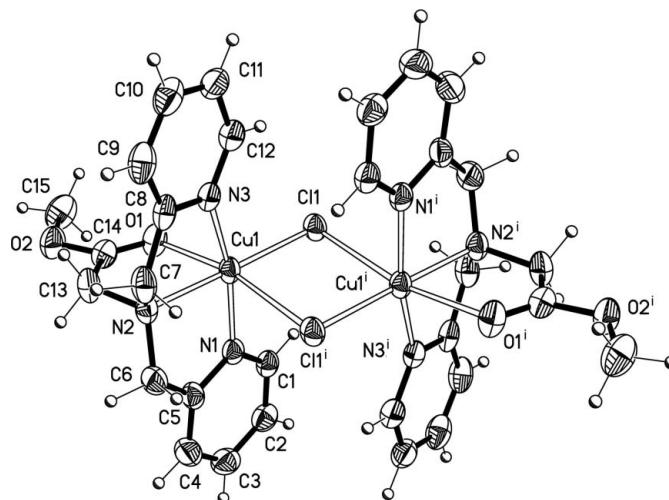


Figure 1

The cationic complex in (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. [Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, -z$.]

and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for Csp^2 , $\text{C}-\text{H} = 0.99$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the methylene groups, and $\text{C}-\text{H} = 0.98$ Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl group. The methyl group was also allowed to rotate about its local threefold axis. The O atom of the water molecule is located on a twofold rotation axis at $(\frac{1}{2}, y, \frac{1}{4})$. The unique H atom was placed initially along the $\text{O1W} \cdots \text{O2A}$ vector then refined with the $\text{O1W}-\text{H1W}$ distance restrained to be 0.84 (1) Å, the $\text{H1W} \cdots \text{H1W}^{\text{ii}}$ distance restrained to be 1.37 (1) Å [symmetry code: (ii) $1 - x, y, \frac{1}{2} - z$], and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The largest peak in the difference density lies in the vicinity of the perchlorate anion.

Data collection: APEX2 (Bruker–Nonius, 2004); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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