Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Aqua(1*H*-imidazole-κN^3)(triethanolamine-$\kappa^3 O$)copper(II) perchlorate hemi(benzene-1,4-dioxyacetate)

Shan Gao,[a]* Ji-Wei Liu,[a] Jian-Rong Li,[b] Li-Hua Huo[a] and Hui Zhao[a]

[a]College of Chemistry and Chemical Technology, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail:
shangao67@yahoo.com

Received 25 November 2003
Accepted 12 December 2003
Online 19 December 2003

Key indicators

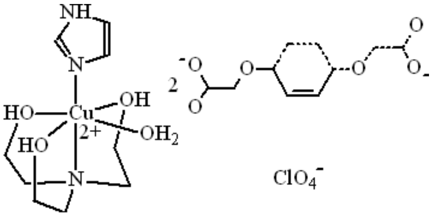
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 15.8

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

The asymmetric unit of the title novel copper(II) complex, $[Cu(C_6H_{15}NO_3)(C_3H_4N_2)(H_2O)](ClO_4)(C_{10}H_8O_6)_{0.5}$, is composed of a Cu^{II} complex cation with triethanolamine and imidazole ligands, a perchlorate anion and half of a benzene-1,4-dioxyacetate dianion. In the complex cation, the Cu^{II} ion is six-coordinate, with a distorted N_2O_4 octahedral geometry. A supramolecular network structure is formed by intermolecular hydrogen bonds.

Comment

Interesting structures and properties have been reported for several triethanolamine complexes. In the course of these structural investigations, it has been found that triethanolamine is a polydentate ligand, functioning as either a tetradentate (Naiini *et al.*, 1994, 1997) or a tridentate ligand (Krabbes *et al.*, 1999, 2000; Sanni *et al.*, 1987). Here, we report the crystal structure of the title novel copper(II) complex, $[Cu(triethanolamine)(imidazole)(H_2O)](ClO_4)(C_{10}H_8O_6)_{0.5}$, (I), which incorporates this ligand.



(I)

As shown in Fig. 1, the crystal structure of (I) reveals the asymmetric unit to consist of a mononuclear Cu^{II} complex cation, $[Cu(triethanolamine)(imidazole)(H_2O)]^{2+}$, and a perchlorate and half of a benzene-1,4-dioxyacetate as anions. The triethanolamine ligand is bonded to the Cu^{II} ion in a tetradentate chelating mode. A Cu^{II} complex with the triethanolamine ligand has been reported previously (Krabbes *et al.*, 1999), but there the ligand coordinated in a tridentate chelating mode.

Selected bond distances and angles for (I) are given in Table 1. The Cu^{II} atom is coordinated by hydroxyl groups O1, O2 and O3, and atom N1 of the triethanolamine ligand, atom N2 of the imidazole ligand and one water molecule, so forming a distorted octahedral coordination geometry. The Cu—O3 bond distance in the equatorial plane is 2.010 (2) Å, which is much shorter than the two long apical Cu—O bonds to the triethanolamine ligand [2.321 (2) and 2.339 (2) Å]. The Cu—N(triethanolamine) bond is 2.042 (2) Å, which is slightly longer than the Cu—N(imidazole) bond of 1.954 (2) Å. The

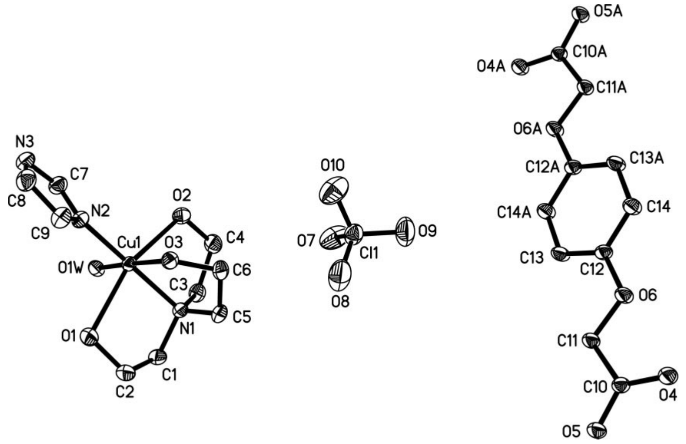


Figure 1
The individual ions of (I), with displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (A) $-x, -1 - y, 1 - z$.]

cis O—Cu—O bond angles around the central Cu^{II} atom vary from 78.10 (8) to 98.02 (8)°. In addition, a supramolecular framework is formed through intermolecular hydrogen bonds (Table 2).

Experimental

The title complex was prepared by the addition of triethanolamine (7 ml), imidazole (20 mmol) and benzene-1,4-dioxyacetic acid (20 mmol) to an aqueous solution of copper perchlorate (20 mmol). The mixture was filtered and blue prismatic single crystals of (I) were obtained from the solution at room temperature over several days.

Crystal data

$[Cu(C_6H_{15}NO_3)(C_3H_4N_2)(H_2O)]$-$(ClO_4)(C_{10}H_8O_6)_{0.5}$
M_r = 510.36
Triclinic, $P\overline{1}$
a = 8.629 (2) Å
b = 10.059 (2) Å
c = 13.254 (3) Å
α = 94.48 (3)°
β = 95.15 (3)°
γ = 114.49 (3)°
V = 1034.2 (5) Å^3
Z = 2
D_x = 1.639 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4246 reflections
θ = 3.1–27.3°
μ = 1.25 mm^{-1}
T = 293 (2) K
Prism, blue
0.36 × 0.26 × 0.21 mm

Data collection

Rigaku R-AXIS Rapid diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.662, T_{max} = 0.780
7437 measured reflections
4659 independent reflections
3908 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.4°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.06
4659 reflections
295 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.3927P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N2	1.954 (2)	Cu1—N1	2.042 (2)
Cu1—O3	2.010 (2)	Cu1—O2	2.321 (2)
Cu1—O1*W*	2.021 (2)	Cu1—O1	2.339 (2)
N2—Cu1—O3	91.23 (8)	O1*W*—Cu1—O2	85.18 (7)
N2—Cu1—O1*W*	93.44 (8)	N1—Cu1—O2	78.10 (8)
O3—Cu1—O1*W*	174.84 (7)	N2—Cu1—O1	98.02 (8)
N2—Cu1—N1	173.88 (7)	O3—Cu1—O1	96.66 (7)
O3—Cu1—N1	83.15 (7)	O1*W*—Cu1—O1	84.85 (7)
O1*W*—Cu1—N1	92.26 (7)	N1—Cu1—O1	80.28 (8)
N2—Cu1—O2	104.59 (8)	O2—Cu1—O1	155.77 (7)
O3—Cu1—O2	91.53 (7)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H19*A*···O5^{i}	0.803 (17)	1.954 (18)	2.735 (2)	164 (3)
N3—H15···O7^{ii}	0.863 (18)	2.10 (2)	2.935 (3)	162 (3)
O1*W*—H19*B*···O5^{iii}	0.816 (17)	1.886 (18)	2.695 (2)	171 (3)
O2—H18···O4^{iii}	0.820 (17)	1.871 (17)	2.690 (2)	177 (3)
O3—H17···O4^{iv}	0.799 (17)	1.958 (19)	2.732 (2)	163 (3)
O3—H17···O6^{iv}	0.799 (17)	2.47 (3)	2.931 (2)	118 (2)
O1—H16···O9^{v}	0.813 (18)	2.30 (2)	3.092 (4)	166 (4)
O1—H16···O7^{v}	0.813 (18)	2.49 (3)	3.117 (4)	135 (3)

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

H atoms attached to O and N atoms were located from Fourier difference maps and their parameters were refined isotropically. The H atoms attached to C atoms were included in calculated positions and treated as riding atoms, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *RAPID AUTO* (Rigaku, 1998); cell refinement: *RAPID AUTO*; data reduction: *CrystalStructure* (Molecular Structure Corporation and Rigaku, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

The work was supported by the National Natural Science Foundation of China (grant No. 20101003) and Heilongjiang province Natural Science Foundation (grant No. B0007).

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Acta Cryst. (2004). E**60**, m94–m95 S. Gao *et al.* • $[Cu(C_6H_{15}NO_3)(C_3H_4N_2)(H_2O)](ClO_4)\cdot0.5C_{10}H_8O_6$ **m95**