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Bis{μ-2-[bis(2-hydroxyethyl)amino]ethanolato}-bis(4-methylbenzoato)dicopper(II) dihydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.086
Data-to-parameter ratio = 16.9

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

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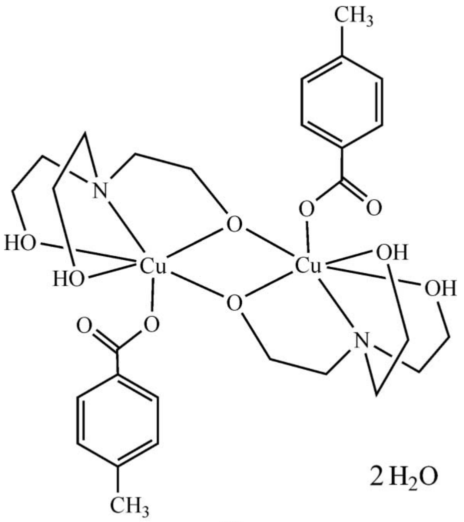
The title compound, $[Cu_2(C_8H_7O_2)_2(C_6H_{14}NO_3)_2]\cdot 2H_2O$, contains a centrosymmetric binuclear $[Cu_2(H_2tea)_2]^{2+}$ fragment (H_3tea is triethanolamine), with two bound 4-methylbenzoate anions. All O atoms of H_2tea, one O atom of 4-methylbenzoate and two solvent water molecules are involved in O—H···O hydrogen bonding, providing additional stabilization of the binuclear molecular units and also linking them into one-dimensional hydrogen-bonded chains.

Comment

The title compound, (I), has been previously obtained (Kirillov *et al.*, 2006) and applied as a catalyst or catalyst precursor for the mild peroxidative oxidation of cyclohexane within our ongoing research on the synthesis and catalytic properties of various Cu (Kirillov, Kopylovich, Kirillova, Haukka *et al.*, 2005; Karabach *et al.*, 2006), Re (Kirillov, Haukka *et al.*, 2005) and Ni (Haukka *et al.*, 2005) complexes derived from triethanolamine (H_3tea). Compound (I) has been characterized previously by FT–IR, FAB$^+$–MS and elemental analysis. We report here its X-ray crystal structure.



(I)

The crystal structure of (I) (Fig. 1) consists of binuclear $[Cu_2(H_2tea)_2]^{2+}$ units linked to two monodentate 4-methylbenzoate anions. Each Cu^{II} centre has a distorted tetragonal–bipyramidal geometry and each H_2tea acts as a tetradentate ligand with one bridging alkoxo-group generating a centrosymmetric planar $Cu_2(\mu\text{-}O)_2$ core, in which the Cu···Cu separation is 2.9070 (7) Å. Most of the bonding parameters of (I) (Table 1) are comparable to those of related copper

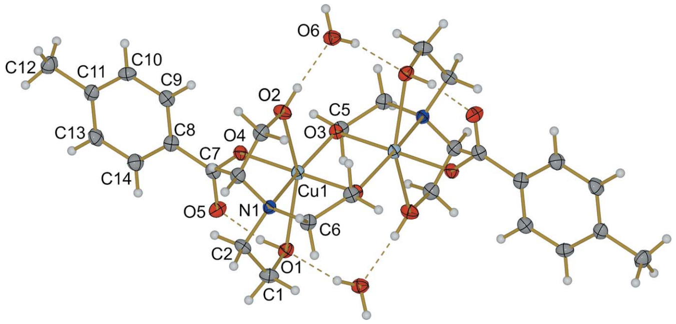


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. Unlabelled atoms are related to labelled atoms by the symmetry operator $(1 - x, -y, 2 - z)$. Dashed lines denote hydrogen bonds.

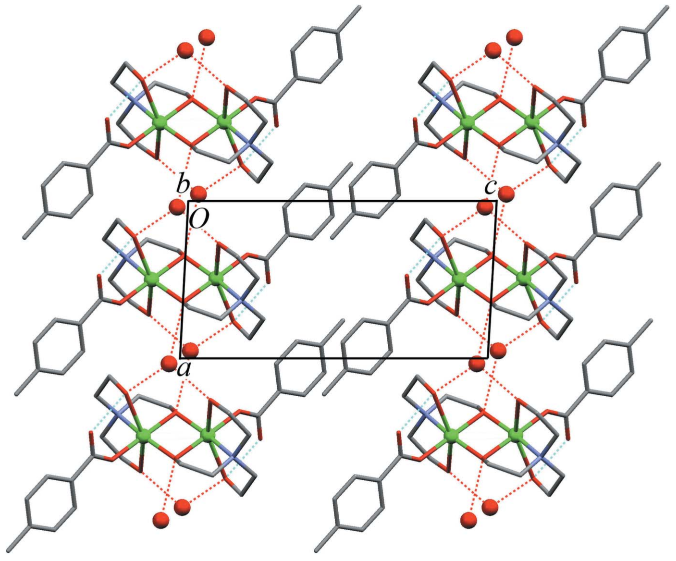


Figure 2
View of (I) along the *b* axis, showing the O—H···O hydrogen-bonding network (dashed lines). H atoms are omitted. O atoms of the solvent water molecules are shown as red spheres.

triethanolaminate dimeric or polymeric complexes in which the $[Cu_2(H_2tea)_2]^{2+}$ unit is linked to benzoate and terephthalate (Kirillov, Kopylovich, Kirillova, Haukka *et al.*, 2005), 3-chlorobenzoate (Kirillov *et al.*, 2006), saccharinate (Topcu *et al.*, 2002) or *trans*-1,2-bis(4-pyridyl)ethylene (Tudor *et al.*, 2003).

In (I), the binding of the 4-methylbenzoate anion is additionally stabilized by intramolecular O1—H10···O5 hydrogen bonds (Fig. 1, Table 2) between an OH group of H_2tea and the free carboxylate oxygen, forming an almost planar six-membered O1/H10/O5/C7/O4/Cu1 ring. Each solvent water molecule acts as both a hydrogen-bond acceptor and donor (Table 2), bridging between triethanolaminate OH groups, providing further reinforcement of the binuclear units (Fig. 1). The water molecules also interact with the μ-O atoms of H_2tea in neighbouring molecular units, forming one-dimensional hydrogen-bonded polymers running along the *a* axis (Fig. 2).

Experimental

The title compound was obtained according to the previously described procedure (Kirillov *et al.*, 2006). Pale-blue crystals were formed in a few days upon evaporation of the aqueous reaction mixture in air at ambient temperature.

Crystal data

$[Cu_2(C_8H_7O_2)_2(C_6H_{14}NO_3)_2]\cdot 2H_2O$
$M_r = 729.75$
Triclinic, $P\overline{1}$
$a = 7.5137$ (5) Å
$b = 7.9384$ (5) Å
$c = 14.3761$ (10) Å
$\alpha = 75.422$ (3)°
$\beta = 87.976$ (4)°
$\gamma = 70.805$ (4)°
$V = 782.69$ (9) Å^3
$Z = 1$
$D_x = 1.548$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 1.42$ mm^{-1}
$T = 120$ (2) K
Plate, pale blue
0.17 × 0.15 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: numerical (*XPREP*; Bruker, 2005)
$T_{min} = 0.794$, $T_{max} = 0.944$
12377 measured reflections
3429 independent reflections
2743 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.086$
$S = 1.06$
3429 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2 + 0.6222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.66$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	2.5249 (18)	Cu1—O3^i	1.9497 (17)
Cu1—O2	2.4535 (18)	Cu1—O4	1.9491 (18)
Cu1—O3	1.9443 (17)	Cu1—N1	2.051 (2)
O3—Cu1—O4	95.09 (7)	N1—Cu1—O2	76.44 (7)
O3—Cu1—O3^i	83.42 (7)	O3—Cu1—O1	114.12 (6)
O3—Cu1—N1	161.95 (8)	O4—Cu1—O1	88.55 (7)
O4—Cu1—N1	96.98 (8)	N1—Cu1—O1	79.62 (7)
O3—Cu1—O2	91.36 (7)	O2—Cu1—O1	154.18 (6)
O4—Cu1—O2	84.90 (7)	Cu1—O3—Cu1^i	96.58 (7)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O5	0.91	1.70	2.594 (3)	168
O2—H2O···O6^i	0.87	1.82	2.680 (3)	174
O6—H6O···O1	0.82	2.02	2.824 (3)	165
O6—H6P···O3^{ii}	0.90	2.07	2.891 (2)	151

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

H atoms bound to C atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.95–0.99 Å and $U_{iso} = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for the methyl group. H atoms bound to O atoms were located in difference Fourier maps, then constrained to ride on their parent atoms in their as-found positions (distances given in Table 2), with $U_{iso}(H) = 1.5U_{eq}(O)$.

Acta Cryst. (2007). E**63**, m526–m528 Kirillov *et al.* • $[Cu_2(C_8H_7O_2)_2(C_6H_{14}NO_3)_2]\cdot 2H_2O$ **m527**

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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