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A trinuclear copper complex, $[Cu_3L_2](ClO_4)_2$, where H_2L is 1,3-bis(2-hydroxybenzoylimino)propane

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Key indicators

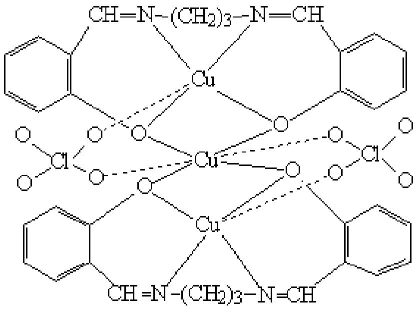
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.053
wR factor = 0.110
Data-to-parameter ratio = 16.4

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

The crystal structure determination of the title complex, bis[μ-1,3-bis(2-oxybenzoylimino)propane]tricopper(II) diperchlorate, $[Cu_3(C_{17}N_2H_{16}O_2)_2](ClO_4)_2$, shows that a Cu^{II} atom, located on a crystallographic inversion centre, is coordinated by four bridging O atoms of two *L* ligands [H_2L is 1,3-bis(2-hydroxybenzoylimino)propane] and two perchlorate O atoms, in elongated octahedral geometry. The other two Cu^{II} atoms are in a distorted square-pyramidal geometry, ligated by N and O atoms of the *L* ligand and an O atom of a perchlorate ion. In the complex, the Cu—N bond length is 1.948 (4) Å and the Cu—O bond lengths are in the range 1.930 (3)–1.984 (3) Å.

Comment

Several Schiff base derivatives of salicylaldehyde, pyridine aldehyde and imidazole aldehyde and amines containing one, two and three amino groups are known (Colacio *et al.*, 1998; Elder, 1978; Long *et al.*, 1999; Mimura *et al.*, 1998; Root *et al.*, 1993; Roslyn *et al.*, 1985;Yang *et al.*, 2000; Yang *et al.*, 2001). These compounds are used as ligands in the complexation reaction of various cations. Recently we have synthesized a series of imidazole-containing Schiff base compounds, which can be used in crystal engineering or in the synthesis of transition metal complexes to obtain structural models of some metalloenzymes (Long *et al.*, 1999; Yang, Chen & Ji, 2000; Yang, Tong *et al.*, 2001). As a continuation of our studies, we have isolated the title compound, (I), and report here its preparation and structure.



(I)

The crystal structure of (I) consists of a trinuclear $[Cu_3(L_2)]^{2+}$ cation [where H_2L = 1,3-bis(2-hydroxybenzoylimino)propane], and two perchlorate anions. A view of the coordination environment of the trinuclear $[Cu_3(L_2)]^{2+}$ cation is shown in Fig. 1. A perspective drawing of the molecular packing in the unit cell is shown in Fig. 2. In the trinuclear cation, atom Cu2, located on a crystallographic inversion

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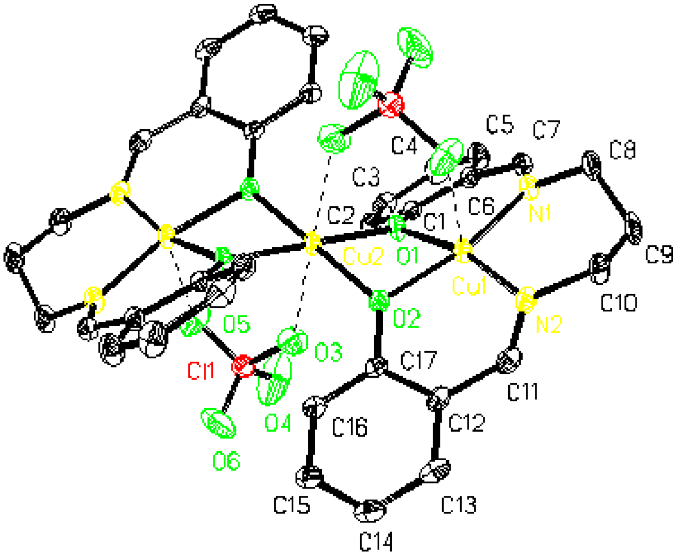

Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level.

centre, is coordinated by four bridging O atoms of the two ligands in the equatorial positions, and the axial positions are occupied by two perchlorate O atoms at much longer distances [Cu···O = 2.488 (4) Å]. Thus, the central Cu atom adopts highly elongated octahedral geometry due to the Jahn–Teller effect (Cotton & Wilkinson, 1972), which is very commonly observed in Cu^{II} complexes. The other two Cu^{II} atoms in the complex are coordinated by two N and two O atoms of the *L* ligand in the basal positions of a distorted square pyramid, with the apical position occupied by one perchlorate O atom at a much longer distance [Cu···O = 2.600 (4) Å]. The Cu—N bond length is 1.948 (4) Å and the Cu—O bond lengths are in the range 1.930 (3)–1.984 (3) Å, which are typical values for Cu^{II} complexes.

Experimental

To a solution of 1,3-diaminopropane (5 mmol, 0.375 g) in dried CH_3OH (25 ml) was added a solution of salicylaldehyde (10 mmol, 1.221 g) in dried CH_3OH (30 ml). The mixture was refluxed for 2.5 h to afford a yellow solution. The addition of a solution of copper acetate (8.0 mmol, 1.44 g) and sodium perchlorate (5.0 mmol, 0.60 g) in aqueous methanol (20 ml, 1:1 *v*/*v*) gave an immediate blue precipitate. The solid material was removed by filtration and the solution left to stand for several days. The blue crystals of the complex that separated from solution were collected and washed with methanol.

Crystal data

$[Cu_3(C_{17}N_2H_{16}O_2)_2](ClO_4)_2$
$M_r = 950.19$
Monoclinic, $P2_1/n$
$a = 8.8249$ (10) Å
$b = 11.9200$ (13) Å
$c = 17.2030$ (19) Å
$\beta = 103.161$ (2)°
$V = 1762.1$ (3) Å^3
$Z = 2$

$D_x = 1.791$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4098 reflections
$\theta = 2$–28.3°
$\mu = 2.02$ mm^{-1}
$T = 293$ (2) K
Plate, blue
0.15 × 0.10 × 0.05 mm

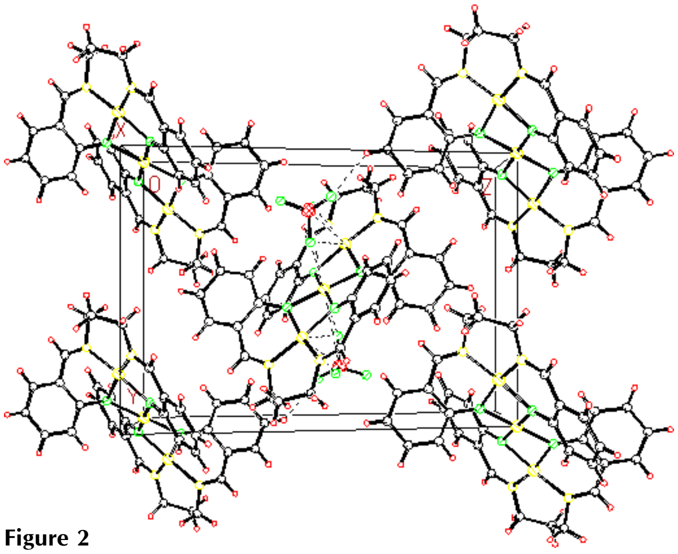

Figure 2
A perspective view (Farrugia, 1997) of the crystal packing of (I), viewed down the *a* axis.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.788$, $T_{max} = 0.899$
10495 measured reflections

4099 independent reflections
2716 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 28.3°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 15$
$l = -22 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.110$
$S = 0.96$
4099 reflections
250 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.62$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.935 (3)	Cu1···Cu2	2.9446 (5)
Cu1—O2	1.944 (3)	Cu2—O1	1.930 (3)
Cu1—N2	1.948 (4)	Cu2—O2	1.984 (3)
Cu1—N1	1.948 (4)		
O1—Cu1—O2	77.93 (12)	O1^i—Cu2—O1	180
O1—Cu1—N2	165.97 (14)	O1^i—Cu2—O2	102.91 (12)
O2—Cu1—N2	92.62 (14)	O1—Cu2—O2	77.09 (12)
O1—Cu1—N1	90.98 (14)	O2—Cu2—O2^i	180
O2—Cu1—N1	167.22 (14)	Cu1^i···Cu2···Cu1	180
N2—Cu1—N1	99.40 (16)		

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

All H atoms were positioned geometrically (C—H = 0.96 Å) and refined using the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$ of the parent atom. Two O atoms of the perchlorate anion (O4 and O6) exhibit larger displacement ellipsoids indicating disorder. However, no suitable disorder model was found. In the perchlorate anion, the Cl—O and O···O distances were restrained to be 1.40 (2) and 2.32 (2) Å, respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998) and *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL97*.

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