

***trans*-Bis(ethanolamine-*N,O*)bis-(saccharinato-*N*)copper(II)**Veysel T. Yilmaz,^{a*} Omer Andac,^a Yildiray Topcu^a and William T. A. Harrison^b^aDepartment of Chemistry, Faculty of Arts and Science, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, and ^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland
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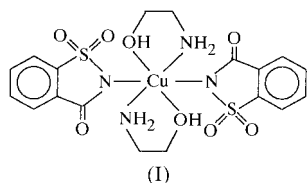
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In the title complex, $[\text{Cu}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_2\text{H}_7\text{NO})_2]$, the Cu^{II} centre lies on an inversion centre and exhibits octahedral coordination, with the two ethanolamine (Hea) and two saccharinate [sac; anionic 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide] ligands in a *trans* configuration. The bidentate Hea ligands bridge axial and equatorial positions and the sac anions occupy equatorial sites around the distorted octahedral copper(II) centre [$\text{Cu}-\text{O} = 2.3263(16)$, $\text{Cu}-\text{N}_{\text{Hea}} = 1.9923(16)$ and $\text{Cu}-\text{N}_{\text{sac}} = 2.1776(16)$ Å].

Comment

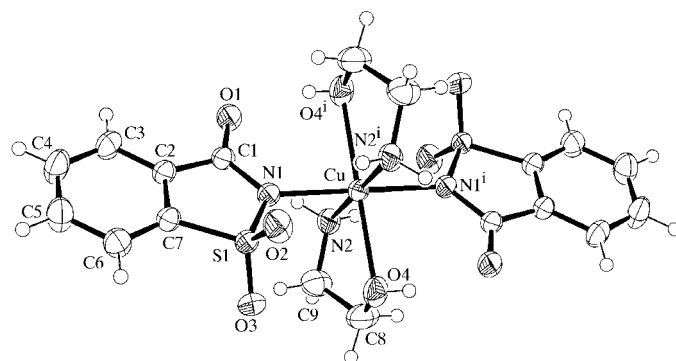
Saccharin (*o*-sulfobenzimide) is widely used as a non-calorific artificial sweetener. The chemistry of its mixed-ligand metal complexes is of interest owing to their potential effectiveness in biological systems, which is due to the coordination ability of saccharin towards most metals present in body fluid (Haider *et al.*, 1985). It has also been suggested that the importance of saccharin lies in its potential use as an antidote for metal poisoning (Ainscough *et al.*, 1990). In light of this interest, we have prepared the title compound, (I), and present its crystal structure here.



In compound (I), the complex lies on an inversion centre and is octahedrally coordinated to two saccharinate (sac) ions and two ethanolamine (Hea) molecules. The Hea molecules act as bidentate ligands and form two five-membered *trans*-chelate rings which bridge axial and equatorial sites, the O-donor being axial and the N-donor equatorial, while each sac anion behaves as a monodentate ligand occupying an equatorial position (Fig. 1).

The $\text{Cu}-\text{O}$ and $\text{Cu}-\text{N}_{\text{Hea}}$ distances of 2.3263 (16) and 1.9923 (16) Å, respectively, are typical for $\text{Cu}-\text{Hea}$ complexes (Hursthouse *et al.*, 1990; Bombicz *et al.*, 1997). The sac ligand is essentially planar and intramolecular bond lengths are virtually identical to those found in the free saccharin molecule (Okaya, 1969). The $\text{Cu}-\text{N}_{\text{sac}}$ distance of 2.1776 (16) Å is significantly longer than in related Cu^{II} complexes, *cf.* 2.030 (5) Å in $[\text{Cu}(\text{sac})_2(\text{bpy})_2]\text{sac}\cdot 2\text{H}_2\text{O}$ (Hergold-Brundić *et al.*, 1991) and 2.032 (2) Å in $[\text{Cu}(\text{H}_2\text{O})(\text{py})_2(\text{sac})_2]$ (Jovanovski *et al.*, 1998).

The H atoms of the hydroxyl and amino groups of the Hea molecules participate in both intra- and intermolecular hydrogen bonding with the carbonyl and sulfonyl O atoms of the sac ligand (Table 2). The phenyl H6 atom of the sac ligand appears to be involved in an intermolecular hydrogen bond with the carbonyl O1 atom of a sac ligand in a neighbouring molecule related by a *C*-glide operation.

**Figure 1**

The structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small spheres of arbitrary radii [symmetry code: (i) $-x, -y, 1-z$].

Experimental

Previously prepared $[\text{Cu}(\text{sac})_2(\text{H}_2\text{O})_4]\cdot 2\text{H}_2\text{O}$ (1.07 g, 2.0 mmol) was dissolved with stirring in ethanol (50 ml) at 343 K and the solution was cooled to room temperature. Ethanolamine (0.25 g, 4.0 mmol) was then added dropwise to the solution. The resulting dark-blue solution was left at room temperature until evaporation resulted in the formation of blue crystals of (I) suitable for X-ray diffraction analysis.

Crystal data

$[\text{Cu}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_2\text{H}_7\text{NO})_2]$
 $M_r = 550.06$
 Monoclinic, $P2_1/c$
 $a = 11.0346(5)$ Å
 $b = 8.8957(4)$ Å
 $c = 11.4161(5)$ Å
 $\beta = 103.946(1)^\circ$
 $V = 1087.58(8)$ Å³
 $Z = 2$

$D_x = 1.680$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4688 reflections
 $\theta = 2.93\text{--}29.98^\circ$
 $\mu = 1.251$ mm⁻¹
 $T = 298(2)$ K
 Block, blue
 $0.32 \times 0.24 \times 0.17$ mm

Data collection

Bruker SMART1000 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{\text{min}} = 0.667$, $T_{\text{max}} = 0.808$
 9396 measured reflections

3165 independent reflections
 2524 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 30.01^\circ$
 $h = -15 \rightarrow 15$
 $k = -11 \rightarrow 12$
 $l = -16 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.099$
 $S = 1.040$
 3165 reflections
 154 parameters
 H atoms: see text

$$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 0.0557P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.82 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cu—N1	2.1776 (16)	Cu—O4	2.3263 (16)
Cu—N2	1.9923 (16)		
N1—Cu—N2	89.26 (7)	N2—Cu—O4	80.97 (6)
N1—Cu—O4	92.76 (5)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4O \cdots O1 ⁱ	0.76 (3)	1.92 (3)	2.650 (2)	160 (3)
N2—H2B \cdots O2 ⁱ	0.90	2.42	3.214 (2)	148
N2—H2A \cdots O3 ⁱⁱ	0.90	2.38	3.169 (2)	147
C6—H6 \cdots O1 ⁱⁱⁱ	0.93	2.46	3.261 (2)	144

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

The hydroxyl-group H atom was located from a difference map and freely refined, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The other H atoms were positioned geometrically at distances of 0.90, 0.93 and 0.97 \AA for amino, methylene/methyl and aromatic H atoms, respectively, and refined riding on their parent atoms.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1435). Services for accessing these data are described at the back of the journal.

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