

Bis(triethanolamine-*N,O,O'*)zinc(II) disaccharinateYildiray Topcu,^a Veysel T. Yilmaz^{a*} and Carsten Thöne^b^aDepartment of Chemistry, Faculty of Arts and Science, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, and ^bInstitut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

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Key indicators

Single-crystal X-ray study

T = 133 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.040

wR factor = 0.113

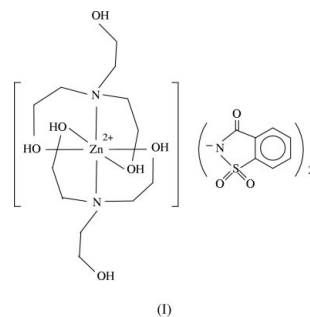
Data-to-parameter ratio = 19.7

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

The structure of the title complex, $[\text{Zn}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_7\text{H}_4\text{NO}_3\text{S})_2$, consists of a complex cation and two saccharinate (sac) anions. In the complex cation, the zinc(II) ion is octahedrally coordinated by two tridentate (*N,O,O'*) triethanolamine (tea) ligands with a facial distribution. Two ethanol groups of each tea ligand form two five-membered chelate rings around the zinc(II) ion, while the third ethanol group remains uncoordinated. In addition to weak $\pi-\pi$ interactions between the phenyl rings of neighbouring sac ions, H atoms of free and coordinated hydroxyl groups are involved in hydrogen bonding with amine N, carbonyl O and sulfonyl O atoms of the adjacent sac ions, forming a three-dimensional network.

Comment

This work is a part of our extensive research on the synthesis, spectral, thermal and structural characterization of mixed ligand complexes containing the saccharinate ion (sac), chemically known as *o*-sulfobenzimide, and ethanolamines such as monoethanolamine (Yilmaz, Andac *et al.*, 2001; Yilmaz, Topcu *et al.*, 2001; Andac *et al.*, 2001), monoethanol-ethylenediamine (Yilmaz, Andac *et al.*, 2001), diethanolamine (Yilmaz, Topcu *et al.*, 2001) and triethanolamine (İcbudak *et al.*, 1995; Topcu *et al.*, 2001). In order to extend this investigation to the 2B metal complexes, we report here the crystal and molecular structure of the complex of zinc(II) saccharinate with triethanolamine (tea), (I).



The structure of the title complex, (I), consists of a complex cation, $[\text{Zn}(\text{tea})_2]^{2+}$, and two sac anions. In the cation, the Zn atom sits on an inversion centre and is six-coordinated by two tea molecules in a distorted octahedral geometry (Fig. 1). Each tea ligand acts as a tripodal and tridentate ligand through the N and two hydroxyl O atoms, forming two five-membered bidentate chelate rings around the zinc(II) ion. The other hydroxyl group of the tea ligand is not involved in coordination. The two tea ligands occupy the *cis* positions of the

Received 30 October 2001

Accepted 19 November 2001

Online 30 November 2001

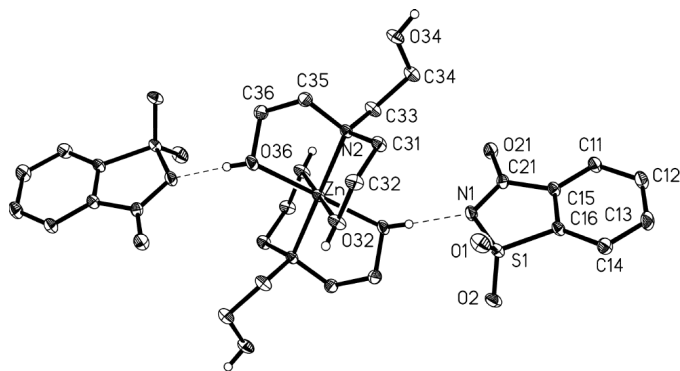


Figure 1

The molecular structure of $[\text{Zn}(\text{tea})_2](\text{sac})_2$ (50% displacement ellipsoids). H atoms are drawn as small spheres of arbitrary radii.

octahedron with a facial configuration. The Zn–N and Zn–O bond distances compare well with the corresponding bond distances of Zn–N = 2.0873 (9) Å and Zn–O = 2.1642 (8) Å in $[\text{Zn}(\text{ea})_2(\text{sac})_2]$ (Yilmaz, Karadag & Thoene, 2001). The interatomic distances within the tea ligand are in agreement with literature values (İcbudak *et al.*, 1995; Topcu *et al.*, 2001). Some distortion of regular octahedral geometry is apparent as shown in Table 1.

Both sac ions are essentially planar, with an r.m.s. deviation of 0.03 Å, and the bond geometry of the sac ion is similar to those of reported complexes containing sac as the counter-ion (Deng *et al.*, 2000; Topcu *et al.*, 2001; Yilmaz, Yilmaz *et al.*, 2001). The packing of the title compound exhibits parallel stacking of pairs of the sac ions as shown in Fig. 2. Thus, the phenyl rings approximately superimpose on each other and are connected by weak π – π interactions of 3.996 Å. The H atom of the free hydroxyl group of the tea ligand forms a hydrogen bond with the carbonyl O atom of the sac ions [H34...O2ⁱⁱ 2.06 (2) Å; symmetry code: (i) $x - 1, y + 1, z$], while the coordinated hydroxyl H atoms are involved in intermolecular hydrogen bonding with the carbonyl O and the negatively charged amine N atoms of the sac ions [H32...O2ⁱ 1.84 (2) Å and H36...N1ⁱⁱⁱ 1.97 (2) Å; symmetry codes: (i) $1 + x, y, z$; (iii) $1 - x, 1 - y, 1 - z$]. The overall hydrogen-bond scheme can be described as a three-dimensional network formed by the tea ligands and sac anions.

Experimental

Previously prepared $[\text{Zn}(\text{sac})_2(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$ (0.54 g, 1.0 mmol) was dissolved in 40 ml of a methanol–2-propanol mixture (1:1) at 333 K with stirring and cooled to room temperature. Then tea (0.30 g, 2.0 mmol) was added to the solution dropwise. The resulting solution was left to stand at room temperature and allowed to evaporate slowly over a few days for crystallization. The colourless crystals suitable for X-ray diffraction analysis were collected by suction filtration, washed with acetone and dried in air.

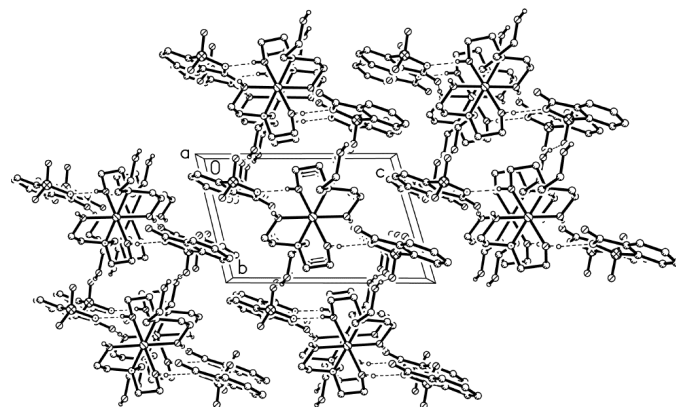


Figure 2

Packing diagram of $[\text{Zn}(\text{tea})_2](\text{sac})_2$ showing the hydrogen-bonding scheme.

Crystal data

$[\text{Zn}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_7\text{H}_4\text{NO}_3\text{S})_2$
 $M_r = 728.09$
 Triclinic, $P\bar{1}$
 $a = 7.9428$ (12) Å
 $b = 8.6647$ (14) Å
 $c = 12.1383$ (18) Å
 $\alpha = 75.190$ (6)°
 $\beta = 87.432$ (6)°
 $\gamma = 65.639$ (6)°
 $V = 734.0$ (2) Å³

$Z = 1$
 $D_x = 1.647$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 8018 reflections
 $\theta = 2.6$ – 30.5 °
 $\mu = 1.05$ mm⁻¹
 $T = 133$ (2) K
 Tablet, colourless
 $0.34 \times 0.15 \times 0.08$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
 ω and φ scans
 Absorption correction: multi-scans (SADABS; Bruker, 1998)
 $T_{\min} = 0.716$, $T_{\max} = 0.921$
 12 918 measured reflections

4266 independent reflections
 3731 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$
 $\theta_{\max} = 30.0$ °
 $h = -11 \rightarrow 11$
 $k = -12 \rightarrow 12$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.113$
 $S = 1.06$
 4266 reflections
 217 parameters

H-atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0814P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.17$ e Å⁻³
 $\Delta\rho_{\min} = -1.81$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C11–C16	1.377 (2)	Zn–N2	2.1037 (14)
C11–C12	1.392 (2)	Zn–O32	2.1555 (11)
C12–C13	1.388 (2)	Zn–O36	2.1573 (13)
C13–C14	1.388 (2)	N2–C31	1.4841 (19)
C14–C15	1.385 (2)	N2–C35	1.492 (2)
C15–C16	1.385 (2)	N2–C33	1.4953 (19)
C15–C21	1.494 (2)	C31–C32	1.512 (2)
C16–S1	1.7585 (16)	C32–O32	1.434 (2)
C21–O21	1.2385 (18)	C33–C34	1.510 (2)
C21–N1	1.351 (2)	C34–O34	1.414 (2)
S1–O1	1.4332 (13)	C35–C36	1.507 (2)
S1–O2	1.4484 (13)	C36–O36	1.440 (2)
S1–N1	1.6151 (14)		
N2 ⁱ –Zn–O32	99.00 (5)	O32–Zn–O36 ⁱ	85.66 (5)
N2–Zn–O32	81.00 (5)	N2–Zn–O36	81.21 (5)
N2–Zn–O36 ⁱ	98.79 (5)	O32–Zn–O36	94.34 (5)

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O32—H32 \cdots O21 ⁱ	0.823 (18)	1.844 (19)	2.6580 (16)	170 (3)
O34—H34 \cdots O2 ⁱⁱ	0.807 (19)	2.06 (2)	2.8174 (17)	157 (3)
O36—H36 \cdots N1 ⁱⁱⁱ	0.780 (19)	1.97 (2)	2.7453 (18)	171 (3)

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

H atoms bonded to C atoms were included using a riding model, starting from calculated positions. Other H atoms were refined freely.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

We thank Ondokuz Mayıs University for the financial support given to this research project.

References

- Andac, O., Topcu, Y., Yilmaz, V. T. & Harrison, W. T. A. (2001). *J. Chem. Crystallogr.* In the press.
- Bruker (1998). *SMART* (Version 5.0), *SAINT* (Version 4.0) and *SADABS* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
- Deng, R. M. K., Bilton, C., Dillon, K. B. & Howard, J. A. K. (2000). *Acta Cryst. C* **56**, 142–145.
- İçbudak, H., Yilmaz, V. T., Howie, R. A., Andac, Ö. & Ölmez, H. (1995). *Acta Cryst. C* **51**, 1759–1761.
- Sheldrick, G. M. (1990). *Acta Cryst. A* **46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Wisconsin, Madison, USA.
- Topcu, Y., Andac, O., Yilmaz, V. T. & Harrison, W. T. A. (2001). *Acta Cryst. E* **57**, m82–m84.
- Yilmaz, V. T., Andac, O., Topcu, Y. & Harrison, W. T. A. (2001). *Acta Cryst. C* **57**, 271–272.
- Yilmaz, V. T., Karadag, A. & Thoenes, C. J. (2001). *Coord. Chem.* In the press.
- Yilmaz, V. T., Topcu, Y., Yilmaz, F. & Thoenes, C. (2001). *Polyhedron*, **20**, 3209–3217.
- Yilmaz, V. T., Yilmaz, F., Topcu, Y., Andac, O. & Guven, K. (2001). *J. Mol. Struct.* **560**, 9–13.