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Acta Cryst. (1997). C**53**, 54–56

Aquabis(salicylaldoximato)zinc

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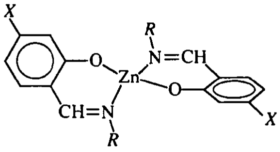
(*Received 25 April 1996; accepted 16 September 1996*)

Abstract

The crystal structure of aqua(2-hydroxybenzaldehyde oximato-*N*,*O*)zinc(II), $[Zn(C_7H_6NO_2)_2(H_2O)]$, is formed by discrete molecules. The coordination polyhedron of the Zn atom is a distorted trigonal bipyramid with a ZnN_2O_3 chromophore.

Comment

As far as the structures of zinc complexes of type (1) are known, the vast majority are monomeric with a tetrahedral N_2O_2 coordination of zinc: *R*† = Et, Pr (*X* = OMe), ^{i}Pr, Bu and $(CH_2)_3SEt$ (Dreher, Elias & Paulus, 1987); *R* = Ph and 4-MePh (Xia-Lan, Hong-Li & Fang-Ming, 1992); *R* = 4-MePh (Sogo *et al.*, 1988); *R* = CH(Ph)Me (Sakiyama, Okawa, Matsumoto & Kida, 1990); *R* = 4-MeOPh (*X* = heptoxy) (Polishchuk *et al.*, 1991); *R* = 2,2,6,6-tetramethyl-1-oxypiperidine-4-yl (Gusejnova & Mamedov, 1978); *R* = 2-pyridyl (Vasilchenko *et al.*, 1994). The methyl derivative (*R* = Me) is the only example of a dimeric complex with a Zn_2O_2 unit and distorted trigonal bipyramidal N_2O_3-coordinated Zn atoms (Orioli, Di Vaira & Sacconi, 1966). We report here on the structure of the salicylaldoximato (sald) complex (*R* = OH) which is monomeric but has a five-coordinated Zn atom as a result of solvation.



(1)

The title structure consists of a racemic mixture of two stereoisomers. The labelling scheme of the molecule is shown in Fig. 1. The Zn atom is five-coordinated by two sald anions and one water molecule (in the equatorial plane). The coordination polyhedron is described by a distorted trigonal bipyramid. In this trigonal bipyramid, the two axial Zn—O1 and Zn—O11 bonds are of practically the same length [2.041 (4) and 2.047 (4) Å, respectively] and are almost collinear [O1—Zn—O11 172.70 (15)°]. The two equatorial Zn—N1 and Zn—N11 bonds have the same lengths as the axial Zn—O bonds [2.040 (5) and 2.042 (5) Å, respectively]. The third equatorial Zn—O3 distance of 1.942 (4) Å is significantly shorter than the other two. The out-of-plane angles lie within the range 87.4 (2)–94.0 (2)°, whereas the bond angles in the equatorial plane lie within the range 117.0 (2)–122.8 (2)°. The sum of the bond angles in the equatorial plane (360.0°) indicates coplanarity of the Zn atom with the three

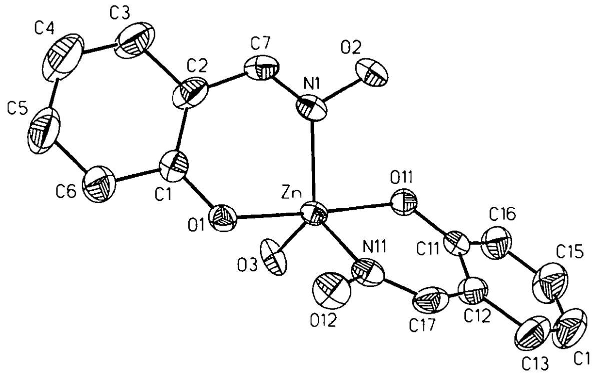


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound showing the labelling of the atoms. Displacement ellipsoids are plotted at the 40% probability level.

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† *X* = H unless otherwise stated.

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coordinated atoms [deviation of the Zn atom from the O3—N1—N11 plane is 0.006 (1) Å towards the O1 atom]. The sald ligands are coordinated both in equatorial and axial positions. Both sald ligands are almost planar, the largest atomic deviation from the corresponding mean plane is 0.093 (5) Å. The mean planes of the two sald ligands are inclined at an angle of 45.3 (1)° with respect to one another.

Within the molecule, strong O12···O1 and O2···O11 intramolecular hydrogen bonds are observed [2.778 (8) and 2.749 (5) Å, respectively].

In the structure, the molecules of the title compound are connected *via* strong O3···O1^{i} and O3···O11^{ii} intermolecular hydrogen bonds [2.679 (6) and 2.650 (8) Å, respectively] to form infinite chains. These chains are linked together by substantially weaker O2···O2^{iii} and O12···O12^{iv} hydrogen bonds [3.042 (10) and 2.986 (6) Å, respectively] [symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, y+\frac{1}{2}, \frac{3}{2}-z$; (iii) $\frac{1}{2}-x, -y-\frac{1}{2}, 1-z$; (iv) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$]. In this way, isolated layers are formed.

A similar structure to the complex described [(1), *R* = OH] is exhibited by the aqua adduct of bis(salicylidene)-1,2-diaminoethane–zinc, [Zn(salen)(H_2O)] (Hall & Moore, 1966). The trigonal bipyramid observed in this complex is much more distorted than that in (1) (*R* = OH).

Experimental

Crystals of the title compound were prepared by mixing a water/ethanol solution of $ZnAc_2$ with an ethanol solution of salicylaldoxime (saldH). White (later brownish yellow) light-sensitive crystals of the title complex appeared within a few days.

Crystal data

[Zn($C_7H_6NO_2$)$_2$(H_2O)]
M_r = 355.64
Monoclinic
*C*2/*c*
a = 30.136 (9) Å
b = 6.902 (2) Å
c = 16.920 (6) Å
β = 120.23 (2)°
V = 3040.7 (17) Å^3
Z = 8
D_x = 1.554 Mg m^{-3}
D_m = 1.50 (1) Mg m^{-3}
D_m measured by flotation in chloroform–bromoform

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 92 reflections
θ = 10.0–14.1°
μ = 1.639 mm^{-1}
T = 293 (2) K
Fragment
0.49 × 0.30 × 0.19 mm
Brownish yellow

Data collection

Stoe Stadi-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (ψ scans, 10 reflections; North, Phillips & Mathews, 1968)
T_{min} = 0.631, T_{max} = 0.732
2959 measured reflections
2672 independent reflections

2178 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0220
θ_{max} = 25°
h = −35 → 35
k = −8 → 1
l = −20 → 20
3 standard reflections
frequency: 60 min
intensity decay: 4.4%

Refinement

Refinement on F^2
$R(F)$ = 0.0526
$wR(F^2)$ = 0.1746
S = 1.171
2649 reflections
255 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 24.4078P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.040
$\Delta\rho_{max}$ = 0.966 e Å^{-3}
$\Delta\rho_{min}$ = −0.439 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Zn	0.24765 (2)	0.01590 (8)	0.65033 (4)	0.0316 (2)
N1	0.1932 (2)	−0.1340 (6)	0.5400 (3)	0.0370 (11)
O1	0.19993 (14)	0.2500 (5)	0.6018 (2)	0.0365 (9)
O2	0.2083 (2)	−0.3120 (6)	0.5203 (3)	0.0495 (11)
O3	0.2508 (2)	0.0174 (7)	0.7679 (3)	0.0473 (12)
C1	0.1495 (2)	0.2475 (8)	0.5473 (4)	0.0376 (13)
C2	0.1226 (2)	0.0895 (10)	0.4909 (5)	0.052 (2)
C3	0.0693 (3)	0.1032 (14)	0.4343 (8)	0.085 (3)
C4	0.0432 (4)	0.2637 (15)	0.4322 (8)	0.102 (4)
C5	0.0687 (3)	0.4199 (14)	0.4877 (8)	0.087 (3)
C6	0.1206 (3)	0.4127 (11)	0.5437 (6)	0.059 (2)
C7	0.1470 (2)	−0.0895 (9)	0.4852 (4)	0.0435 (14)
N11	0.3006 (2)	0.1663 (6)	0.6333 (3)	0.0380 (11)
O11	0.29489 (15)	−0.2200 (5)	0.6831 (3)	0.0383 (9)
O12	0.2855 (2)	0.3468 (6)	0.5888 (3)	0.0489 (11)
C11	0.3458 (2)	−0.2160 (9)	0.7175 (4)	0.0393 (13)
C12	0.3717 (2)	−0.0557 (9)	0.7070 (4)	0.0447 (15)
C13	0.4250 (3)	−0.0678 (13)	0.7444 (6)	0.069 (2)
C14	0.4524 (3)	−0.2257 (15)	0.7919 (7)	0.087 (3)
C15	0.4273 (3)	−0.3810 (15)	0.8030 (7)	0.082 (3)
C16	0.3746 (3)	−0.3769 (11)	0.7666 (5)	0.059 (2)
C17	0.3466 (3)	0.1216 (10)	0.6592 (4)	0.0450 (15)

Table 2. *Selected geometric parameters (Å, °)*

Zn—O3	1.942 (4)	Zn—N11	2.042 (5)
Zn—N1	2.040 (5)	Zn—O11	2.047 (4)
Zn—O1	2.041 (4)		
O3—Zn—N1	122.8 (2)	O1—Zn—N11	89.0 (2)
O3—Zn—O1	94.0 (2)	O3—Zn—O11	93.3 (2)
N1—Zn—O1	87.4 (2)	N1—Zn—O11	88.5 (2)
O3—Zn—N11	120.2 (2)	O1—Zn—O11	172.70 (15)
N1—Zn—N11	117.0 (2)	N11—Zn—O11	87.5 (2)

Intensities were corrected for Lorentz and polarization factors using *X-RED* (Stoe & Cie, 1995). The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1985) and subsequent Fourier syntheses using *SHELXL*93 (Sheldrick, 1993). Anisotropic displacement parameters were refined for all non-H atoms. All H atoms were located from a difference Fourier map and refined with free isotropic displacement parameters. Geometrical analysis was performed using *PARST* (Nardelli, 1983) and *SHELXL*93. There is a pseudo-twofold axis passing through the Zn atoms approximately parallel to the *c* axis. It brings one ligand (sald) into the other, however, with significant atomic displacements varying from 0.25 to 0.31 Å. In addition, the water molecules are unaffected by this local pseudosymmetry, which excludes any canonical transformation of the monoclinic unit cell into a higher crystal system.

Data collection: Stadi-4 software. Cell refinement: Stadi-4 software. Data reduction: *X-RED*. Program(s) used to solve structure: *SHELXS*86. Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

IP gratefully acknowledges support from the DAAD and Literárny fond Slovenskej republiky. The authors would like to thank the Fonds der Chemischen Industrie for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1207). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 56–58

Tribenzyltin Chloride

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(*Received 11 July 1996; accepted 18 September 1996*)

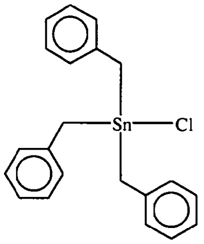
Abstract

The crystal structure of tribenzyltin chloride consists of [$SnCl(C_7H_7)_3$] molecules linked axially by a tin–chlorine bridge [Sn—Cl 2.387 (2), Sn···Cl 3.531 (2) Å and Cl—Sn···Cl 180°] into a linear chain along the *c* axis.

Comment

Among the triorganotin halides, the fluorides universally exist as bridged polymers, whereas the heavier bromides and iodides generally crystallize as monomeric solids (Smith, 1981; Harrison, 1994). The coordination number of tin in the chlorides lies between those in the fluorides and bromides.

The crystal structure of tribenzyltin chloride, (I), consists of $(C_6H_5CH_2)_3SnCl$ molecules (Fig. 1) linked axially by a tin–chlorine bridge [Sn—Cl 2.387 (2), Sn···Cl 3.531 (2) Å and Cl—Sn···Cl 180°] into a linear chain along the *c* axis.



(I)

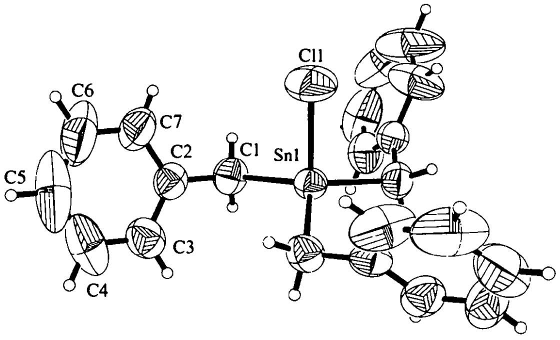


Fig. 1. *ORTEP* [(Johnson, 1965) in *SHELXTL/PC* (Sheldrick, 1990*b*)] plot of tribenzyltin chloride with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

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