Instrumental Achievements

Crystal Structure of [Triaqua(salicylato)(nicotinamide)zinc(II)] Salicylate

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Nicotinamide (NA) is one form of niacin. A deficiency of this vitamin leads to a loss of copper from the body, known as pellagra disease. The nicotinamide derivative, *N,N*-diethylnicotinamide (DENA), is an important respiratory stimulant. A structure determination of the title compound was undertaken in order to determine the ligand properties of NA and salicylate ligands.

The title compound was prepared from the reaction of Zn(2-HO-$C_6H_5COO)_2\cdot 8(H_2O)$ (1.46 g, 3.00 mmol) and NA (0.18 g, 1.50 mmol) in water (80 ml). The mixture was filtered and set aside for crystallization at ambient temperature for a few days.

The results of an X-ray structure determination are given in Tables 1 – 4.

The title compound contains a mononuclear Zn(II) complex in which there are three water molecules, one NA ligand and one salicylate ligand. The one salicylate moiety in the compound does not act as a ligand, but is incorporated into the crystal lattice by hydrogen bonds.

Although the zinc atom has five coordination, close contact of the O5 atom [Zn···O5 = 2.687(6)Å] may cause the zinc atom to have six coordination. The five coordination around Zn(II) can be described as a distorted trigonal bipyramid or a distorted square pyramid.

Further information can be obtained by estimating the structural index, τ,[1] which represents the relative amount of

Table 1 Crystal and experimental data

Formula:	$[ZnO_7N_2C_{13}H_{17}]\cdot C_7O_3H_5$
Formula weight:	515.78
Crystal system:	monoclinic
Space group:	$C2/c$ $Z = 8$
a =	19.600(1)Å
b =	7.518(1)Å
c =	30.918(1)Å
β =	106.45(2)°
V =	4369.1(3)Å^3
D_x =	1.568 g/cm^3
μ(Cu Kα) =	2.09 mm^{-1}
T =	293 K
Crystal color:	colorless
Crystal size:	0.13 × 0.25 × 0.30 mm
λ(Cu Kα) =	1.54184 Å
R = 0.061	wR = 0.068
No. of reflections measured =	4449
No. of reflections used =	2308
[F >3.0 $\sigma(F)$]	
No. of parameters =	323
Goodness-of-fit =	0.91
$(\Delta/\sigma)_{max}$ =	0.01
$(\Delta\rho)_{max}$ =	0.90
$(\Delta\rho)_{min}$ =	–0.58
$2\theta_{max}$ =	148.7°
Measurements:	Enraf-Nonius CAD-4 diffractometer
Program system:	CAD-4 EXPRESS Software
Structure determination:	MolEN
Refinement:	full matrix least-squares
Hydrogen atoms:	difference synthesis and geometric calculation, only the parameters of H6 atom are refined.

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	B_{eq}/Å^2
Zn	0.70613(4)	0.0616(1)	0.55272(3)	3.28(2)
O1	0.7108(2)	0.3383(7)	0.5528(2)	4.0(1)
O2	0.6603(2)	0.0747(7)	0.6030(2)	3.9(1)
O3	0.6890(2)	-0.2156(7)	0.5540(2)	4.1(1)
O4	0.6868(2)	0.0430(7)	0.4863(2)	3.7(1)
O5	0.5796(2)	0.1102(8)	0.4913(2)	4.5(1)
O6	0.4703(2)	0.1157(8)	0.4208(2)	4.8(1)
O7	0.9320(2)	-0.1833(9)	0.5020(2)	5.1(1)
O8	1.0941(3)	-0.054(1)	0.7165(2)	7.6(2)
O9	1.1414(3)	-0.1466(8)	0.6503(2)	5.1(1)
O10	1.2065(3)	0.0405(8)	0.6235(2)	4.8(1)
N1	0.8159(3)	0.0337(8)	0.5808(2)	3.7(1)
N2	1.0350(3)	-0.152(1)	0.5548(2)	4.9(2)
C1	0.6189(3)	0.060(1)	0.4680(2)	3.5(1)
C2	0.5883(3)	0.020(1)	0.4202(2)	3.4(1)
C3	0.6296(3)	-0.053(1)	0.3942(2)	3.9(1)
C4	0.6017(4)	-0.092(1)	0.3491(2)	4.7(2)
C5	0.5303(4)	-0.060(1)	0.3289(2)	5.5(2)
C6	0.4884(4)	0.009(1)	0.3533(3)	5.1(2)
C7	0.5155(3)	0.046(1)	0.3985(2)	4.0(2)
C8	0.8541(3)	-0.035(1)	0.5551(2)	3.5(1)
C9	0.9279(3)	-0.052(1)	0.5709(2)	3.2(1)
C10	0.9600(4)	-0.001(1)	0.6144(3)	4.8(2)
C11	0.9205(4)	0.070(1)	0.6405(3)	5.2(2)
C12	0.8486(4)	0.086(1)	0.6224(2)	4.4(2)
C13	0.9649(3)	-0.134(1)	0.5393(2)	3.5(1)
C14	1.1736(3)	0.002(1)	0.6515(2)	3.8(2)
C15	1.1691(4)	0.132(1)	0.6865(2)	4.2(2)
C16	1.1297(4)	0.101(1)	0.7168(3)	5.5(2)
C17	1.1270(5)	0.226(2)	0.7481(3)	8.0(3)
C18	1.1633(6)	0.387(2)	0.7507(3)	8.4(3)
C19	1.2027(5)	0.420(1)	0.7214(3)	7.2(3)
C20	1.2051(4)	0.294(1)	0.6899(3)	5.1(2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

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Table 3 Bond distances (Å) and angles (˚)

Zn-O1	2.082(5)	Zn-O4	1.986(5)
Zn-O2	2.006(5)	Zn-N1	2.089(5)
Zn-O3	2.113(5)		
O1-Zn-O2	88.8(2)	O2-Zn-O4	144.0(2)
O1-Zn-O3	173.0(2)	O2-Zn-N1	108.5(2)
O1-Zn-O4	93.8(2)	O3-Zn-O4	88.0(2)
O1-Zn-N1	93.4(2)	O3-Zn-N1	92.5(2)
O2-Zn-O3	85.7(2)	O4-Zn-N1	107.2(2)

Table 4 Torsion angles (˚)

C3-C2-C1-O4	5.97(1.1)	O4-Zn-N1-C12	161.6(6)
C2-C1-O4-Zn	-168.9(5)	O7-C13-C9-C8	-0.4(1.1)
C1-O4-Zn-N1	-179.9(5)	O10-C14-C15-C20	-3.5(1.1)

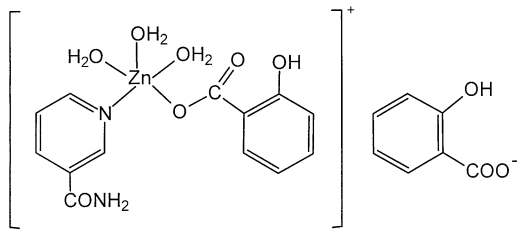

Fig. 1 Chemical diagram.

trigonality (square pyramid, τ0; trigonal pyramid, τ1; $\tau(\beta - \alpha)/60^\circ$, α and β being the two largest angles [βO1–Zn–O3 173.0(2)˚ and αO2–Zn–O4 144.0(2)˚] around the central atom.

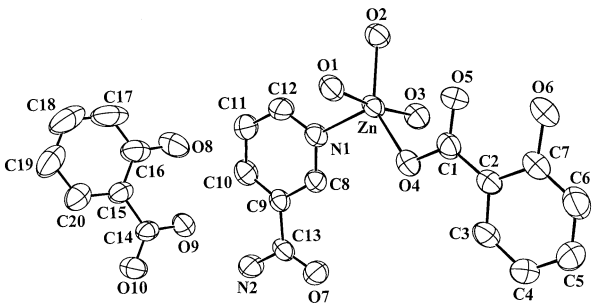

Fig. 2 Molecular structure of the title compound with the atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

The value of τ is 0.48. The co-ordination geometry of the Zn(II) atom is therefore best described as a distorted trigonal bipyramidal.

There are intramolecular hydrogen bondings between O6–H6···O5 [O5···O6 2.588(7), H6···O5 1.672(12)Å, O6–H6···O5 141.4(13)˚] and O8–H8′···O9 [O8···O9 2.571(11), H8′···O9 1.782 Å, O8–H8′···O9 133.6˚]. The configuration around the Zn atom is given by the torsion angles (Table 4). The Zn atom is out of the C1, O4, O5 plane by –0.208(1)Å. The dihedral angle between the carboxyl group and the phenyl ring is 6.53 (1.04)˚.

References

1. S. Uhlenbrock, R. Wegner, and B. Krebs, *J. Chem. Soc. Dalton Trans*., **1996**, 3731.