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Synthesis and Crystal Structure of a New Oxime-Type Zinc(II) Complex

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The Zn(II) complex of an oxime has been synthesized by the reaction of 1-(4-{[(E)-3-ethoxy-2-hydroxybenzylidene]amino}phenyl)ethanone oxime and zinc(II) acetate dihydrate in the mixture of ethanol and acetonitrile. The molecule crystallizes in the triclinic system, space group P-1. Each zine(II) atom coordinates with two mono-oxime ligands in tetrahedral coordination. In the crystal structure, each molecule connects with two other molecules through two strong intermolecular N4-H4···O1 hydrogen bonds and two N2-H2···O4 intermolecular hydrogen bonds. Moreover, a 1D supramolecular structure formed with another adjacent molecule by the two pairs of intermolecular N1-H1···O1 and N2-H2···O4 hydrogen bonds.

Key Words: Oxime-type compound, Synthesis, Crystal structure, Zine(II) complex.

INTRODUCTION

Oxime-type compounds have been playing an important role in the development of coordination chemistry[1,2]. During the past several decades, a large number of oxime-type complexes have been studied extensively for easily tunable steric position and application[3]. Many oxime-type compounds have been structurally characterized[4], but only a relatively small number of oxime-type complexes have been characterized[5-7]. Here, we report the synthesis and crystal structure of a new oxime-type Zn(II) complex.

EXPERIMENTAL

The reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory. C, H and N analyses were carried out with a GmbH VariuoEL V3.00 automatic elemental analyzer. IR spectra in the range 4000-400 cm^{-1} were recorded on a VERTEX70 FT-IR spectrophotometer using KBr pellets. X-Ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector. Melting points were measured by the use of a microscopic melting point apparatus made in Beijing Taike Instrument Limited Company and the thermometer was uncorrected.

General procedure: 1-(4-{[(E)-3-ethoxy-2-hydroxybenzylidene]amino}phenyl)ethanone oxime was synthesized according to an analogous method reported earlier[8,9].

Synthesis of Zn(II) complex: To 2 mL ethanol/acetonitrile (1:1) solution of the mono-oxime ligand (2.98 mg, 0.01 mmol) was added the same solution of zinc(II) acetate dihydrate (1.10 mg, 0.005 mmol) and the mixture was stand at room temperature for *ca.* 2 weeks. Yellow block-like single crystals suitable for X-ray diffraction studies were obtained by slow evaporation from a ethanol/acetonitrile (1:1) solution. Yield 75.6 %. Anal. calcd. (%) for $C_{75}H_{84.5}N_{9.5}O_{14}Zn_2$ (%): C, 61.12; H, 5.78; N, 9.03; Zn, 8.87. Found (%): C, 61.40; H, 5.63; N, 9.20; Zn, 8.65.

TABLE-1
CRYSTAL DATA AND REFINEMENT PARAMETERS FOR THE TITLE COMPOUND

Empirical formula	$C_{75}H_{84.50}N_{9.50}O_{14}Zn_2$
Formula weight	1473.76
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Cell dimensions	a = 13.9414(15) Å, b = 17.6058(18) Å, c = 18.278(2) Å, α = 83.775(2), β = 108.232(1), γ = 68.3210(10)
Volume	4052.9(8) $Å^3$
Z	2
Density (calculated)	1.208 mg/m^3
Absorption coefficient	0.655 mm^{-1}
$F_{(000)}$	1546
Index ranges	$-16 \le h \le 8$, $-20 \le k \le 19$, $-21 \le l \le 19$
Reflections collected	20503/14116 [$R_{(int)}$ = 0.0718]
Independent reflections	4682
Data/restraints/parameters	14116/0/988
Goodness of fit indicator	1.037
R [I > 2σ(I)]	R_1 = 0.0700, wR_2 = 0.1491
Largest diff. peak and hole	0.591 and -0.406 e $Å^{-3}$

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR THE TITLE COMPOUND

Bond	Lengths	Bond	Lengths	Bond	Lengths
Zn(1)-O(4)	1.912(4)	N(5)-C(35)	1.299(7)	O(5)-C(21)	1.34(1)
Zn(1)-O(1)	1.923(4)	N(5)-C(44)	1.442(7)	O(5)-C(25)	1.43(1)
Zn(1)-N(3)	1.985(8)	N(6)-C(50)	1.337(8)	O(6)-C(33)	1.227(8)
Zn(1)-N(1)	1.997(6)	N(6)-C(47)	1.404(7)	O(7)-C(37)	1.322(7)
Zn(2)-O(10)	1.912(4)	N(7)-C(52)	1.311(8)	O(8)-C(38)	1.372(9)
Zn(2)-O(7)	1.937(4)	N(7)-C(61)	1.421(8)	O(8)-C(42)	1.344(9)
Zn(2)-N(7)	1.997(5)	N(8)-C(67)	1.334(9)	O(9)-C(50)	1.237(7)
Zn(2)-N(5)	2.001(5)	N(8)-C(64)	1.405(8)	O(10)-C(54)	1.325(7)
N(1)-C(1)	1.490(9)	N(9)-C(77)	1.15(7)	O(11)-C(55)	1.352(8)
N(1)-C(10)	1.371(9)	N(10)-C(71)	1.2(2)	O(11)-C(59)	1.417(8)
N(2)-C(16)	1.394(8)	N(10')-C(71')	1.2(1)	O(12)-C(67)	1.202(8)
N(2)-C(13)	1.367(8)	O(1)-C(3)	1.312(7)	O(13)-C(69)	1.42(1)
N(3)-C(18)	1.387(8)	O(2)-C(4)	1.359(8)	O(14)-C(73)	1.4(1)
N(3)-C(27)	1.372(8)	O(2)-C(8)	1.439(7)	O(15)-C(75)	1.44(6)
N(4)-C(33)	1.374(8)	O(3)-C(16)	1.253(9)	–	–
N(4)-C(30)	1.448(8)	O(4)-C(20)	1.323(8)	–	–
Bond	Angles	Bond	Angles	Bond	Angles
O(4)-Zn(1)-O(1)	116.8(2)	C(11)-C(10)-C(15)	120.6(7)	C(38)-C(39)-C(40)	120.2(8)
O(4)-Zn(1)-N(3)	96.7(2)	C(11)-C(10)-N(1)	117.8(7)	C(41)-C(40)-C(39)	120.4(8)
O(1)-Zn(1)-N(3)	116.2(2)	C(15)-C(10)-N(1)	121.4(7)	C(40)-C(41)-C(36)	120.7(8)
O(4)-Zn(1)-N(1)	113.2(2)	C(10)-C(11)-C(12)	119.8(7)	O(8)-C(42)-C(43)	107.4(8)
O(1)-Zn(1)-N(1)	96.9(2)	C(11)-C(12)-C(13)	119.9(7)	C(45)-C(44)-C(49)	117.4(7)
N(3)-Zn(1)-N(1)	123.1(2)	C(14)-C(13)-N(2)	117.2(7)	C(45)-C(44)-N(5)	120.8(6)
O(10)-Zn(2)-O(7)	115.9(2)	C(14)-C(13)-C(12)	119.0(7)	C(49)-C(44)-N(5)	121.7(6)
O(10)-Zn(2)-N(7)	97.0(2)	N(2)-C(13)-C(12)	123.7(8)	C(44)-C(45)-C(46)	122.9(6)
O(7)-Zn(2)-N(7)	108.3(2)	C(13)-C(14)-C(15)	120.3(7)	C(45)-C(46)-C(47)	119.2(7)
O(10)-Zn(2)-N(5)	110.5(2)	C(10)-C(15)-C(14)	120.3(7)	C(48)-C(47)-C(46)	118.0(7)
O(7)-Zn(2)-N(5)	96.5(2)	O(3)-C(16)-N(2)	122.8(9)	C(48)-C(47)-N(6)	117.9(6)
N(7)-Zn(2)-N(5)	129.9(2)	O(3)-C(16)-C(17)	120(1)	C(46)-C(47)-N(6)	123.9(7)
C(1)-N(1)-C(10)	116.2(6)	N(2)-C(16)-C(17)	117.2(9)	C(49)-C(48)-C(47)	121.8(6)
C(1)-N(1)-Zn(1)	120.1(5)	N(3)-C(18)-C(19)	129.6(8)	C(48)-C(49)-C(44)	120.6(6)
C(10)-N(1)-Zn(1)	123.4(5)	C(20)-C(19)-C(18)	123.7(8)	O(9)-C(50)-N(6)	122.9(7)
C(16)-N(2)-C(13)	129.8(8)	C(20)-C(19)-C(24)	120.5(9)	O(9)-C(50)-C(51)	119.3(7)
C(18)-N(3)-C(27)	119.0(6)	C(18)-C(19)-C(24)	116(1)	N(6)-C(50)-C(51)	117.9(7)
C(18)-N(3)-Zn(1)	119.8(5)	O(4)-C(20)-C(19)	125.3(8)	N(7)-C(52)-C(53)	128.8(7)
C(27)-N(3)-Zn(1)	120.9(4)	O(4)-C(20)-C(21)	117(1)	C(52)-C(53)-C(54)	124.9(7)
C(33)-N(4)-C(30)	129.8(7)	C(19)-C(20)-C(21)	117.6(9)	C(52)-C(53)-C(58)	116.1(7)
C(35)-N(5)-C(44)	119.0(6)	O(5)-C(21)-C(22)	126(1)	C(54)-C(53)-C(58)	119.1(8)
C(35)-N(5)-Zn(2)	120.4(5)	O(5)-C(21)-C(20)	114(1)	O(10)-C(54)-C(55)	118.1(7)
C(44)-N(5)-Zn(2)	120.6(4)	C(22)-C(21)-C(20)	120(1)	O(10)-C(54)-C(53)	124.0(7)
C(50)-N(6)-C(47)	129.9(6)	C(23)-C(22)-C(21)	121(1)	C(55)-C(54)-C(53)	117.9(7)
C(52)-N(7)-C(61)	118.5(6)	C(22)-C(23)-C(24)	122(1)	O(11)-C(55)-C(56)	125.1(8)
C(52)-N(7)-Zn(2)	119.0(5)	C(23)-C(24)-C(19)	120(1)	O(11)-C(55)-C(54)	114.6(7)
C(61)-N(7)-Zn(2)	122.1(5)	O(5)-C(25)-C(26)	109.1(9)	C(56)-C(55)-C(54)	120.3(7)
C(67)-N(8)-C(64)	129.1(7)	C(32)-C(27)-C(28)	117.6(7)	C(57)-C(56)-C(55)	121.2(8)
C(3)-O(1)-Zn(1)	125.1(4)	C(32)-C(27)-N(3)	119.9(7)	C(58)-C(57)-C(56)	120.1(8)
C(4)-O(2)-C(8)	117.2(6)	C(28)-C(27)-N(3)	122.4(7)	C(57)-C(58)-C(53)	121.3(8)
C(20)-O(4)-Zn(1)	124.5(5)	C(29)-C(28)-C(27)	120.2(6)	O(11)-C(59)-C(60)	108.2(7)
C(21)-O(5)-C(25)	118.6(9)	C(28)-C(29)-C(30)	121.8(7)	C(62)-C(61)-C(66)	117.6(8)
C(37)-O(7)-Zn(2)	125.3(5)	C(29)-C(30)-N(4)	117.7(7)	C(62)-C(61)-N(7)	120.0(7)
C(38)-O(8)-C(42)	117.1(7)	C(29)-C(30)-C(31)	117.3(7)	C(66)-C(61)-N(7)	122.1(7)
C(54)-O(10)-Zn(2)	124.0(4)	N(4)-C(30)-C(31)	124.9(7)	C(61)-C(62)-C(63)	123.2(7)
C(55)-O(11)-C(59)	119.5(6)	C(32)-C(31)-C(30)	120.1(7)	C(64)-C(63)-C(62)	120.0(8)
N(1)-C(1)-C(2)	128.4(7)	C(27)-C(32)-C(31)	123.0(7)	C(63)-C(64)-N(8)	125.4(8)
C(3)-C(2)-C(7)	120.0(7)	O(6)-C(33)-N(4)	123.5(9)	C(63)-C(64)-C(65)	116.7(8)
C(3)-C(2)-C(1)	124.1(7)	O(6)-C(33)-C(34)	120.1(8)	N(8)-C(64)-C(65)	117.9(7)
C(7)-C(2)-C(1)	115.8(7)	N(4)-C(33)-C(34)	116.3(8)	C(66)-C(65)-C(64)	122.6(7)
O(1)-C(3)-C(2)	125.2(6)	N(5)-C(35)-C(36)	128.4(7)	C(65)-C(66)-C(61)	119.5(8)
O(1)-C(3)-C(4)	118.1(7)	C(37)-C(36)-C(41)	119.8(7)	O(12)-C(67)-N(8)	122.3(8)
C(2)-C(3)-C(4)	116.7(7)	C(37)-C(36)-C(35)	124.8(7)	O(12)-C(67)-C(68)	121.8(9)
O(2)-C(4)-C(5)	125.1(7)	C(41)-C(36)-C(35)	115.4(7)	N(8)-C(67)-C(68)	115.9(7)
O(2)-C(4)-C(3)	113.3(7)	O(7)-C(37)-C(36)	124.4(7)	O(13)-C(69)-C(70)	113(1)
C(5)-C(4)-C(3)	121.6(8)	O(7)-C(37)-C(38)	118.8(7)	N(10)-C(71)-C(72)	160(1)
C(4)-C(5)-C(6)	121.2(8)	C(36)-C(37)-C(38)	116.7(7)	N(10')-C(71')-C(72')	150(1)
C(7)-C(6)-C(5)	119.1(8)	C(39)-C(38)-O(8)	125.8(9)	O(14)-C(73)-C(74)	115(4)
C(6)-C(7)-C(2)	121.4(8)	C(39)-C(38)-C(37)	122.0(8)	O(15)-C(75)-C(76)	104(4)
O(2)-C(8)-C(9)	107.5(6)	O(8)-C(38)-C(37)	112.2(8)	N(9)-C(77)-C(78)	120(1)

X-Ray structure determination: The single crystal of the present Zn(II) complex, with approximate dimensions of 0.24 mm × 0.14 mm × 0.11 mm was placed on a Bruker Smart 1000 diffractmeter equipped with Apex CCD area detector. The diffraction data were collected using a graphite monochromated MoK_α radition (λ = 0.71073 Å) at 298(2) K. The structure was solved by using the program SHELXS-97 and Fourier difference techniques and refined by full-matrix least-squares method on F^2 using SHELXL-97. Details of the data collection and refinements of the title compound are given in Table-1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically. CCDC: 894712.

RESULTS AND DISCUSSION

X-Ray crystallographic analysis revealed the crystal structure of the title complex. And the structure is shown in Fig. 1. Selected bond distances and angles are listed in Table-2. The molecule crystallizes in the triclinic system, space group P-1 and the unit cell contains two crystallographically independent but chemically identical mononuclear complexs (Fig. 1).

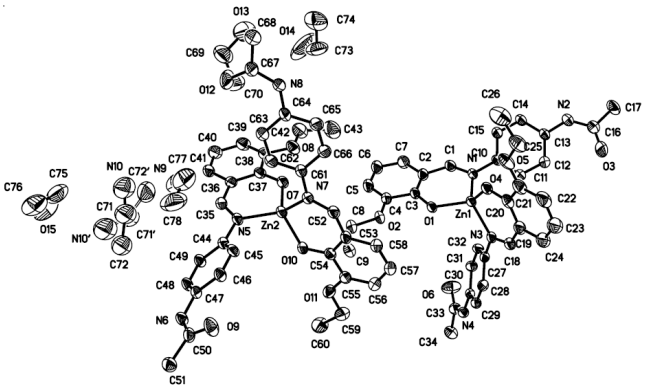

Fig. 1. Molecule structure of the title complex with atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30 % probability level

In the crystal structure, each molecule connects with two other molecules through two strong intermolecular N4-H4···O1 hydrogen bonds and two N2-H2···O4 intermolecular hydrogen bonds. Moreover, a 1D supramolecular structure formed with another adjacent molecule by the two pairs of intermolecular N1-H1···O1 and N2-H2···O4 hydrogen bonds[10-12] (Table-3). The packing arrangement of the unit cell of the title compound is shown in Fig. 2.

TABLE-3
INTRAMOLECULAR AND INTERMOLECULAR O-H···N HYDROGEN-BONDING data [Å, º]

D-H···A	d(D-H)	d(H···A)	d(D···A)	∠D-H···A
N2-H2···O4	0.860	2.14	2.933(3)	154
N2-H2···O5	0.860	1.42	3.076(4)	133
N4-H4···O1	0.860	2.12	2.940(3)	159
N4-H4···O2	0.860	2.45	3.088(3)	132
N6-H6···O10	0.860	2.13	2.941(3)	157
N6-H6···O11	0.860	2.45	3.109(3)	134
N8-H8···O7	0.860	2.21	3.025(3)	158
N8-H8···O8	0.860	2.47	3.137(4)	135
O13-H13···O3	0.820	2.32	3.139(4)	178
O14-H14···O3	0.820	1.81	2.623(4)	171
O15-H15···O6	0.820	2.17	2.944(3)	159

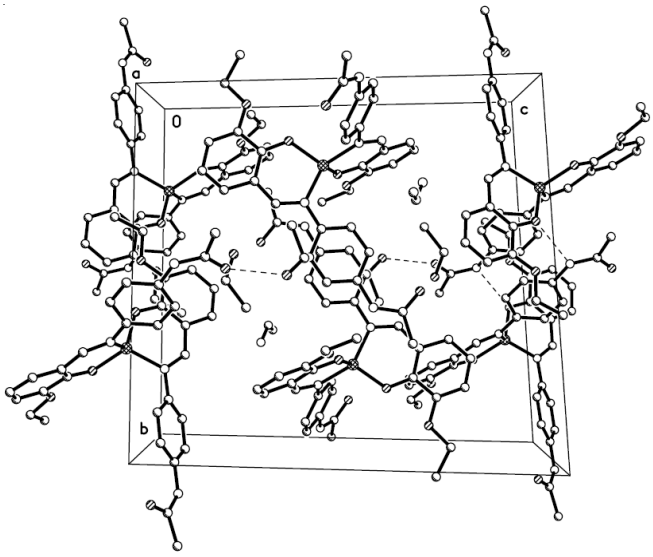

Fig. 2. Packing arrangement of the unit cell of the title compound. H atoms are omitted for clarity

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