Xiao-San Song*

Synthesis and crystal structure of bis{2-bromo-6-((*E*)-((4-((*E*)-1-(methoxy-imino)ethyl)phenyl)imino)methyl)phenolato- $\kappa^2 N, O$ }zinc(II)-methanol(1/2), $C_{65}H_{60}Br_4N_8O_9Zn_2$

<https://doi.org/10.1515/ncrs-2020-0370>

Received July 19, 2020; accepted September 16, 2020;
published online December 14, 2020

Abstract

$C_{65}H_{60}Br_4N_8O_9Zn_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.5707(4)$ Å, $b = 11.1159(5)$ Å, $c = 15.9260(7)$ Å, $\alpha = 92.654(2)^\circ$, $\beta = 104.4460(10)^\circ$, $\gamma = 116.0400(10)^\circ$, $Z = 1$, $V = 1601.90(12)$ Å³, $R_{gt}(F) = 0.0574$, $wR_{ref}(F^2) = 0.1284$, $T = 173(2)$ K.

CCDC no.: 2032293

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

*Corresponding author: Xiao-San Song, School of Environmental and Municipal Engineering, Lanzhou Jiaotong University, Lanzhou, 730070, PR China; and Key laboratory of Yellow River Water Environment in Gansu Province, Lanzhou, 730070, PR China, E-mail: songxs@mail.lzjtu.cn. <https://orcid.org/0000-0003-4522-9143>

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.14 × 0.14 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.31 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 Venture, ω
θ_{max} , completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	17,933, 6290, 0.036
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4763
$N(param)_{refined}$:	447
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

The Schiff base ligand, named 1-{4-[(3-bromo-2-hydroxybenzylidene)amino]phenyl}ethanone *O*-methyl oxime, was prepared by a similar method reported earlier [4–6]. The prepared process of the title complex is as follows: the zinc acetate dihydrate (2.20 mg, 0.01 mmol) dissolved in methanol solution (2 mL) was added dropwise to the prepared ligand (6.95 mg, 0.02 mmol) dissolved in dichloromethane solution (4 mL) at room temperature. After stirring for about 1 h, the mixture was filtered. The filtrate was allowed to stand for 33 days at quiet environment. Several clear light yellow crystals were obtained. **Anal. Calcd.** for $C_{65}H_{60}Br_4N_8O_9Zn_2$: C, 50.45; H, 3.91; N, 7.24. Found: C, 50.83%; H, 4.12%; N, 7.15%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

In recent years, a great number of Zn(II) complexes have been widely investigated due to interesting properties [7,8 and

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.66453 (8)	0.58957 (7)	0.13487 (4)	0.0667 (2)
Br2	1.04954 (6)	0.83614 (6)	0.66248 (4)	0.05193 (17)
Zn1	0.61754 (7)	0.72139 (6)	0.41376 (4)	0.04376 (18)
O1	0.6101 (4)	0.6336 (4)	0.3059 (2)	0.0480 (9)
O2 ^a	0.0544 (7)	0.5668 (6)	0.7866 (4)	0.0772 (19)
O2A ^b	0.020 (3)	0.485 (3)	0.693 (2)	0.0772 (19)
O3	0.7841 (4)	0.7960 (3)	0.5171 (2)	0.0449 (9)
O4 ^c	0.3207 (11)	0.9901 (10)	0.0313 (5)	0.086 (2)
O4A ^d	0.1260 (17)	0.9469 (16)	−0.0255 (10)	0.086 (2)
O5 ^e	0.1622 (12)	0.5354 (11)	0.0053 (6)	0.083 (3)
H5A ^f	0.124207	0.477172	0.035363	0.124*
H5B ^f	0.182292	0.548611	−0.042323	0.124*
N1	0.4220 (5)	0.5812 (4)	0.4221 (3)	0.0444 (11)
N2 ^a	0.1009 (13)	0.5504 (11)	0.7121 (6)	0.069 (2)
N2A ^b	0.113 (5)	0.589 (4)	0.750 (3)	0.069 (2)
N3	0.6102 (4)	0.8970 (4)	0.3978 (3)	0.0411 (10)
N4 ^c	0.1820 (14)	0.9536 (15)	0.0358 (10)	0.079 (3)
N4A ^d	0.225 (3)	0.930 (3)	0.0493 (18)	0.079 (3)
C1	0.4968 (6)	0.5261 (5)	0.2554 (4)	0.0462 (13)
C2	0.3675 (7)	0.4461 (6)	0.2791 (4)	0.0595 (16)
C3	0.2534 (9)	0.3271 (7)	0.2204 (5)	0.093 (3)
H3	0.168533	0.272902	0.237311	0.111*
C4	0.2612 (9)	0.2875 (8)	0.1403 (5)	0.094 (3)
H4	0.182782	0.207159	0.102086	0.112*
C5	0.3862 (8)	0.3669 (7)	0.1152 (4)	0.0706 (19)
H5	0.393777	0.340625	0.059809	0.085*
C6	0.4973 (6)	0.4822 (6)	0.1709 (4)	0.0498 (14)
C7	0.3411 (7)	0.4768 (6)	0.3593 (4)	0.0618 (17)
H7	0.251836	0.411627	0.367559	0.074*
C8	0.3682 (6)	0.5948 (5)	0.4939 (4)	0.0430 (12)
C9	0.2246 (7)	0.5218 (7)	0.4936 (5)	0.077 (2)
H9	0.153332	0.458155	0.442606	0.093*
C10	0.1826 (8)	0.5395 (7)	0.5661 (5)	0.078 (2)
H10	0.081979	0.487189	0.563396	0.094*
C11	0.2793 (6)	0.6295 (5)	0.6425 (4)	0.0466 (13)
C12	0.4218 (7)	0.7038 (6)	0.6418 (4)	0.0664 (18)
H12	0.492285	0.769211	0.692381	0.080*
C13	0.4661 (7)	0.6862 (7)	0.5692 (4)	0.0691 (19)
H13	0.566596	0.738593	0.571615	0.083*
C14	0.2325 (7)	0.6449 (6)	0.7209 (4)	0.0573 (16)
C15	0.3338 (8)	0.7565 (7)	0.7967 (5)	0.0726 (19)
H15A	0.423078	0.747165	0.821320	0.109*
H15B	0.284620	0.751960	0.842013	0.109*
H15C	0.360706	0.844262	0.776997	0.109*
C16 ^a	−0.0726 (14)	0.4448 (13)	0.7845 (9)	0.104 (5)
H16A ^a	−0.144365	0.415967	0.725646	0.156*
H16B ^a	−0.116940	0.460681	0.828206	0.156*
H16C ^a	−0.044067	0.373406	0.798138	0.156*
C16A ^b	−0.090 (6)	0.407 (4)	0.732 (4)	0.093 (18)
H16D ^b	−0.172799	0.331518	0.687623	0.140*
H16E ^b	−0.126261	0.465408	0.755237	0.140*
H16F ^b	−0.048470	0.371253	0.780248	0.140*
C17	0.8602 (5)	0.9242 (5)	0.5540 (3)	0.0385 (11)
C18	0.8229 (5)	1.0288 (5)	0.5275 (3)	0.0405 (12)
C19	0.9117 (6)	1.1639 (5)	0.5737 (4)	0.0494 (14)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
H19	0.884598	1.231919	0.555996	0.059*
C20	1.0354 (6)	1.2000 (6)	0.6430 (4)	0.0562 (15)
H20	1.093529	1.291657	0.673082	0.067*
C21	1.0747 (6)	1.1002 (6)	0.6688 (4)	0.0489 (14)
H21	1.160891	1.123796	0.716436	0.059*
C22	0.9895 (5)	0.9679 (5)	0.6257 (3)	0.0413 (12)
C23	0.7019 (5)	1.0090 (5)	0.4531 (4)	0.0421 (12)
H23	0.687528	1.086366	0.443368	0.051*
C24	0.4982 (5)	0.8993 (5)	0.3264 (3)	0.0412 (12)
C25	0.4059 (6)	0.9493 (6)	0.3415 (4)	0.0495 (14)
H25	0.416152	0.982917	0.400035	0.059*
C26	0.2983 (7)	0.9509 (7)	0.2718 (4)	0.0572 (15)
H26	0.233001	0.982586	0.283178	0.069*
C27	0.2847 (7)	0.9074 (6)	0.1864 (4)	0.0556 (15)
C28	0.3750 (7)	0.8518 (6)	0.1718 (4)	0.0598 (16)
H28	0.364074	0.816988	0.113399	0.072*
C29	0.4803 (7)	0.8473 (6)	0.2419 (4)	0.0529 (14)
H29	0.540100	0.808225	0.231526	0.064*
C30	0.1738 (8)	0.9147 (8)	0.1119 (5)	0.0725 (19)
C31	0.0297 (8)	0.8923 (11)	0.1202 (6)	0.108 (3)
H31A	−0.023387	0.799983	0.131276	0.163*
H31B	−0.028227	0.903432	0.065435	0.163*
H31C	0.044982	0.958584	0.169284	0.163*
C32 ^c	0.3281 (19)	1.0392 (18)	−0.0544 (9)	0.109 (4)
H32A ^c	0.275251	0.961013	−0.102955	0.164*
H32B ^c	0.431492	1.089225	−0.053350	0.164*
H32C ^c	0.282275	1.099376	−0.063024	0.164*
C32A ^d	0.172 (3)	0.959 (3)	−0.0984 (16)	0.109 (4)
H32D ^d	0.115331	0.990072	−0.141721	0.164*
H32E ^d	0.157766	0.869974	−0.123937	0.164*
H32F ^d	0.277332	1.024536	−0.082029	0.164*
C33 ^e	0.075 (4)	0.596 (4)	−0.022 (2)	0.119 (9)
H33B ^f	−0.009263	0.555128	0.000668	0.179*
H33A ^f	0.040557	0.580138	−0.086642	0.179*
H33C ^f	0.130317	0.694048	0.000668	0.179*
C33A ^f	0.055 (3)	0.400 (2)	−0.004 (3)	0.115 (10)
H33D ^f	0.003585	0.356414	−0.066152	0.173*
H33E ^f	0.104375	0.349464	0.025028	0.173*
H33F ^f	−0.016995	0.398654	0.025028	0.173*

^a Occupancy: 0.804(10).^b Occupancy: 0.196(10).^c Occupancy: 0.629(6).^d Occupancy: 0.371(6).^e Occupancy: 0.5.^f Occupancy: 0.25.

many more]. For example: Zn(II) complexes are widely utilized in ion recognition [9, 10], fluorescent material [11, 12] and supramolecular constructing [13–15] fields and so on.

In the title structure Zn1 is located in a N₂O₂-typed coordinated environment, and formed a twisted tetrahedral geometry with coordination atoms. The Zn1–N1 bond length is 2.012(4) Å and the Zn1–N3 is 2.012(4) Å. The Zn1–O1 bond length is 1.903(4) Å and the Zn1–O3 is 1.909(4) Å.

The angles of N1–Zn1–O1 is $97.30(17)^\circ$, N1–Zn1–O3 is $118.84(17)^\circ$, N3–Zn1–O1 is $110.20(17)^\circ$ and O1–Zn1–O3 is $124.66(16)^\circ$, respectively. All geometric parameters are in the typical ranges [16]. This is especially true for the 2-bromo ligand used in this study [4, 17, 18].

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: None declared.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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