Liangjun Lv, Long Lin* and Wei-Na Wu

Crystal structure of bis-[*N*-(3-ethyl-1-pyrazin-2-yl-ethylidene)-3-bromo-benzoic acid-hydrazone- $\kappa^3 O, N, N'$]-cadmium(II), $C_{30}H_{28}N_8O_2Br_2Cd$

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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.15 × 0.11 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.44 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7614, 5213, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3308
$N(\text{param})_{\text{refined}}$:	412
Programs:	SHELX [1], Bruker [2]

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Abstract

$C_{30}H_{28}N_8O_2Br_2Cd$, triclinic, $P\bar{1}$ (no. 2), $a = 8.359(7)$ Å, $b = 12.394(10)$ Å, $c = 15.189(12)$ Å, $\alpha = 95.757(14)^\circ$, $\beta = 90.593(16)^\circ$, $\gamma = 106.961(15)^\circ$, $V = 1496(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0467$, $wR_{\text{ref}}(F^2) = 0.0998$, $T = 296(2)$ K.

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*Corresponding author: Long Lin, School of Materials Science and Engineering, Henan Polytechnic University, Jiaozuo 454000, P.R. China, e-mail: linlong@hpu.edu.cn. <https://orcid.org/0000-0003-1645-5600>

Liangjun Lv: Department of Fundamental Science, Yellow River Conservancy Technical Institute, Kaifeng 475001, P.R. China

Wei-Na Wu: College of Chemistry and Chemical Engineering, Henan Polytechnic University, Jiaozuo 454000, P.R. China. <https://orcid.org/0000-0002-0874-0703>

Source of material

3-Bromo-benzoic acid hydrazide (0.214 g, 1 mmol) and 3-ethyl-2-aceto-pyrazine (0.150 g, 1 mmol) were dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then cadmium(II) acetate (0.166 g, 0.5 mmol) was added. After stirring for 1 h, the mixture was filtered and set aside to crystallize for several days, giving yellow block crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [1]. The C20 and C21 atoms occupied two positions, and the occupancy value of C20/C20A or C21/C21A was refined to 0.61(2) : 0.39(2). The hydrogen atoms were placed at calculated positions and refined using the appropriate SHELXL AFIX riding models.

Comment

As one type of Schiff bases, hydrazones are important an class of ligands with interesting ligation properties due to the presence of several coordination sites, and are widely applied in the field of insecticides, medicines and analytical reagents [3]. In particular, pyrazine-containing hydrazones and their

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cd1	0.22922(6)	0.64530(4)	0.32449(3)	0.04415(15)
Br1	−0.21776(11)	1.02511(6)	0.04826(5)	0.0779(3)
Br2	0.61586(16)	0.79207(9)	0.83608(5)	0.1362(5)
O1	0.0265(5)	0.7240(3)	0.3084(2)	0.0532(11)
O2	0.4052(5)	0.7623(3)	0.4270(2)	0.0562(11)
N1	0.4289(6)	0.5930(4)	0.2364(3)	0.0460(12)
N2	0.6812(6)	0.5528(4)	0.1419(3)	0.0562(14)
N3	0.2450(6)	0.7135(3)	0.1912(3)	0.0364(11)
N4	0.1311(6)	0.7698(4)	0.1727(3)	0.0390(11)
N5	0.0695(6)	0.4508(4)	0.3031(3)	0.0455(12)
N6	−0.0956(9)	0.2275(5)	0.2794(5)	0.090(2)
N7	0.2447(6)	0.5549(4)	0.4452(3)	0.0454(12)
N8	0.3385(6)	0.6172(4)	0.5168(3)	0.0453(12)
C1	0.5246(8)	0.5371(5)	0.2680(4)	0.0589(18)
H1	0.5068	0.5117	0.3237	0.071*
C2	0.6472(9)	0.5170(6)	0.2196(5)	0.066(2)
H2	0.7116	0.4756	0.2423	0.080*
C3	0.5884(7)	0.6121(4)	0.1101(4)	0.0424(14)
C4	0.4569(7)	0.6315(4)	0.1575(3)	0.0366(13)
C5	0.6414(7)	0.6551(5)	0.0233(4)	0.0475(15)
H5A	0.6535	0.7357	0.0281	0.057*
H5B	0.5537	0.6178	−0.0214	0.057*
C6	0.8026(9)	0.6363(6)	−0.0068(4)	0.074(2)
H6A	0.8349	0.6729	−0.0594	0.110*
H6B	0.7875	0.5564	−0.0194	0.110*
H6C	0.8883	0.6677	0.0389	0.110*
C7	0.3371(7)	0.6922(4)	0.1309(3)	0.0351(13)
C8	0.3253(9)	0.7252(6)	0.0403(4)	0.072(2)
H8A	0.2175	0.7357	0.0302	0.109*
H8B	0.3396	0.6665	−0.0024	0.109*
H8C	0.4112	0.7948	0.0345	0.109*
C9	0.0299(7)	0.7701(4)	0.2384(3)	0.0365(13)
C10	−0.0930(7)	0.8334(4)	0.2272(3)	0.0354(13)
C11	−0.0931(7)	0.8931(4)	0.1561(3)	0.0402(14)
H11	−0.0126	0.8969	0.1137	0.048*
C12	−0.2131(8)	0.9471(5)	0.1481(4)	0.0467(15)
C13	−0.3309(8)	0.9448(5)	0.2091(4)	0.0572(17)
H13	−0.4114	0.9821	0.2023	0.069*
C14	−0.3290(8)	0.8871(5)	0.2803(4)	0.0559(17)
H14	−0.4084	0.8853	0.3230	0.067*
C15	−0.2112(7)	0.8313(5)	0.2898(4)	0.0470(15)
H15	−0.2112	0.7919	0.3388	0.056*
C16	−0.0144(9)	0.4041(6)	0.2289(4)	0.0609(18)
H16	−0.0158	0.4483	0.1830	0.073*
C17	−0.0991(10)	0.2922(7)	0.2182(5)	0.082(2)
H17	−0.1611	0.2612	0.1658	0.098*
C18	−0.0091(11)	0.2742(6)	0.3549(5)	0.087(3)
C19	0.0749(7)	0.3883(5)	0.3684(4)	0.0460(15)
C20 ^a	0.0387(17)	0.1811(14)	0.4016(9)	0.076(5)
H20A ^a	0.0648	0.1261	0.3585	0.092*
H20B ^a	0.1347	0.2144	0.4422	0.092*
C21 ^a	−0.106(2)	0.129(2)	0.4483(12)	0.095(7)
H21A ^a	−0.1988	0.0945	0.4068	0.143*
H21B ^a	−0.1328	0.1853	0.4887	0.143*
H21C ^a	−0.0842	0.0718	0.4808	0.143*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C20A ^b	−0.085(3)	0.2066(15)	0.4393(14)	0.064(7)
H20C ^b	−0.0440	0.2528	0.4950	0.077*
H20D ^b	−0.2062	0.1837	0.4367	0.077*
C21A ^b	−0.019(5)	0.106(2)	0.428(2)	0.117(13)
H21D ^b	−0.0599	0.0624	0.3721	0.176*
H21E ^b	−0.0569	0.0591	0.4750	0.176*
H21F ^b	0.1009	0.1310	0.4299	0.176*
C22	0.1733(7)	0.4498(5)	0.4475(4)	0.0418(14)
C23	0.1946(11)	0.3969(5)	0.5277(5)	0.095(3)
H23A	0.2945	0.3736	0.5243	0.142*
H23B	0.0997	0.3319	0.5321	0.142*
H23C	0.2035	0.4507	0.5789	0.142*
C24	0.4112(7)	0.7227(5)	0.4996(4)	0.0415(14)
C25	0.5086(7)	0.7986(5)	0.5744(3)	0.0393(14)
C26	0.5185(8)	0.7647(5)	0.6554(4)	0.0536(17)
H26	0.4670	0.6895	0.6644	0.064*
C27	0.6024(9)	0.8389(6)	0.7240(4)	0.0601(18)
C28	0.6800(8)	0.9513(5)	0.7139(4)	0.0575(17)
H28	0.7370	1.0018	0.7613	0.069*
C29	0.6702(8)	0.9852(5)	0.6328(4)	0.0580(17)
H29	0.7221	1.0605	0.6242	0.070*
C30	0.5857(8)	0.9114(5)	0.5627(4)	0.0509(16)
H30	0.5801	0.9368	0.5075	0.061*

^aOccupancy: 0.61(2), ^bOccupancy: 0.39(2).

metal complexes have been widely investigated mainly due to their excellent biological activities [4–7]. As part of our continuing work, the title complex was synthesized and characterized by X-ray diffraction.

In the title structure, C=O bonds of the hydrazone ligands are enolized, which could be confirmed by the bond lengths of C–O being 1.254(6) and 1.258(6) Å, respectively. The results are in good agreement with previously known acylhydrazone complexes in the literature [4–7]. The central Cd(II) ion is surrounded by two independent anionic ligands with N₂O donor set, thus possessing a distorted octahedral coordination geometry. C–H···O hydrogen bonds form hydrogen-bonded chains that run along the *a*-axis [C14···O2ⁱ = 3.329(7) Å (ⁱ = −1 + *x*, *y*, *z*).

References

- Sheldrick, G. M.: Crystal refinement with SHELX. *Acta Crystallogr.* **C71** (2015) 3–8.
- Bruker. SMART and SAINT. Bruker AXS Inc., Madison, WI, USA (2007).
- Dong, W.-K.; Li, X.-L.; Wang, L.; Zhang, Y.; Ding, Y.-J.: A new application of Salamo-type bisoximes: as a relay-sensor for Zn²⁺/Cu²⁺ and its novel complexes for successive sensing of H⁺/OH[−]. *Sens. Actuators B* **229** (2016) 370–378.
- Xu, J.; Zhou, T.; Xu, Z.-Q.; Gu, X.-N.; Wu, W.-N.; Chen, H.; Wang, Y.; Jia, L.; Zhu, T.-F.; Chen, R.-H.: Synthesis, crystal

- structures and antitumor activities of copper(II) complexes with a 2-acetylpyrazine isonicotinoyl hydrazone ligand. *J. Mol. Struct.* **1128** (2017) 448–454.
5. Wang, W.-W.; Wang, Y.; Zhang, L.; Song, Y.-F.; Wu, W.-N.; Chen, Z.: Syntheses, crystal structures and DNA-binding properties of Cu(II)/Ni(II) complexes with acylhydrazone ligand bearing pyrazine unit. *Chin. J. Inorg. Chem.* **35** (2019) 563–568.
 6. Hou, X.-F.; Zhao, X.-L.; Zhang, L.; Wu, W.-N.; Wang, Y.: Co(II)/Zn(II)/Cu(II) complexes containing hydrazone ligand bearing pyrazine unit: syntheses, crystal structures and fluorescence properties. *Chin. J. Inorg. Chem.* **34** (2018) 201–205.
 7. Huang, W.; Li, Y.; Yong, J.; Liu, Y.; Wu, D.: Hydrazone-based cobalt complexes toward multielectron redox and spin crossover. *RSC Adv.* **8** (2018) 17159–17167.