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The crystal structure of bis(1*H*-benzo[*d*]imidazol-2-amine- κN)-diiodidocadmium(II), $C_{14}H_{14}CdI_2N_6$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.19 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.43 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 PHOTON, φ and ω -scans
$\theta_{\max}$, completeness:	27.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	42428, 4367, 0.037
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3719
$N(\text{param})_{\text{refined}}$:	208
Programs:	Bruker programs [1], SHELX [2], DIAMOND [3]

and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

All the reagents were obtained from commercial sources. A mixture of CdI_2 (0.02 g, 0.05 mmol), 1*H*-benzimidazol-2-amine (0.02 g, 0.15 mmol), *N,N*-dimethylformamide (2 mL) and methanol (2 mL) was placed in a 25 mL Teflon-lined stainless steel vessel and heated at 120 °C for 72 h. After the mixture cooled to room temperature at 5 °C·h⁻¹, crystals of the title complex were obtained.

Comment

In the recent years, the benzimidazoles play an important role in their intriguing structures and potential applications in anticancer activities [4–10]. The 2-position of benzimidazole was replaced by an amino group to get one ligand which has more coordinate sites and different coordinate modes. So we used 1*H*-benzimidazol-2-amine as organic ligand to react with CdI_2 and obtained a new complex.

The asymmetric unit contains one Cd(II) ions, two I^- anions and two 1*H*-benzimidazol-2-amines. Each Cd(II) is coordinated by two iodines (I1, I2) and two nitrogens (N1, N4) from two imidazole rings of the ligands, forming a distorted tetrahedral coordination geometry. The Cd–I bond lengths of 2.7183(4) Å and 2.7367(4) Å and Cd–N bond lengths of 2.218(3) Å and 2.252(3) Å are similar to those reported in $[Cd(2-SC_5H_3NH-3-SiMe_3)_2]_2$ and $[Cd_2(H_{1/2}C_4BIm)_2(CH_3COO)]_n$ (H_3btc = 1,3,5-benzenetricarboxylic acid) [11–13]. The angles around Cd(II) ion range from 100.59(11)° to 112.587(13)°. Some

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Abstract

$C_{14}H_{14}CdI_2N_6$, triclinic, $P\bar{1}$ (no. 2), $a = 7.9247(5)$ Å, $b = 8.9507(5)$ Å, $c = 13.5143(8)$ Å, $\alpha = 92.428(2)^\circ$, $\beta = 92.085(3)^\circ$, $\gamma = 98.871(3)^\circ$, $V = 945.38(10)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0281$, $wR_{\text{ref}}(F^2) = 0.0653$, $T = 298(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions

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

Atom	x	y	z	U _{iso} */U _{eq}
Cd1	0.90320(3)	0.71967(3)	0.25914(2)	0.03950(8)
I1	1.09676(4)	0.54620(3)	0.36016(2)	0.05563(9)
I2	1.06606(4)	0.85039(4)	0.10199(2)	0.06348(10)
N1	0.6483(4)	0.6072(3)	0.2001(2)	0.0407(6)
N2	0.3877(4)	0.5806(4)	0.1303(2)	0.0525(8)
H2A	0.2909	0.6033	0.1104	0.063*
N3	0.5066(4)	0.8216(4)	0.2009(3)	0.0592(9)
H3A	0.5915	0.8790	0.2312	0.071*
H3B	0.4156	0.8577	0.1847	0.071*
N4	0.8279(4)	0.9028(3)	0.3601(2)	0.0448(7)
N5	0.8411(4)	1.1161(4)	0.4525(3)	0.0566(9)
H5A	0.8653	1.2105	0.4706	0.068*
N6	0.9730(5)	1.1296(4)	0.2991(3)	0.0660(10)
H6A	0.9964	1.0843	0.2451	0.079*
H6B	1.0061	1.2254	0.3090	0.079*
C1	0.5162(5)	0.6756(4)	0.1791(3)	0.0439(8)
C2	0.6039(5)	0.4576(4)	0.1615(3)	0.0416(8)
C3	0.6956(6)	0.3387(4)	0.1601(3)	0.0553(10)
H3C	0.8056	0.3496	0.1887	0.066*
C4	0.6181(7)	0.2020(5)	0.1146(4)	0.0674(13)
H4A	0.6776	0.1202	0.1121	0.081*
C5	0.4554(7)	0.1859(5)	0.0733(4)	0.0724(14)
H5B	0.4066	0.0926	0.0441	0.087*
C6	0.3623(6)	0.3024(5)	0.0736(3)	0.0662(12)
H6C	0.2520	0.2901	0.0454	0.079*
C7	0.4398(5)	0.4403(4)	0.1181(3)	0.0484(9)
C8	0.8855(5)	1.0514(4)	0.3675(3)	0.0469(9)
C9	0.7422(5)	0.8707(4)	0.4472(3)	0.0454(8)
C10	0.6590(6)	0.7363(5)	0.4792(3)	0.0588(10)
H10A	0.6532	0.6466	0.4410	0.071*
C11	0.5843(7)	0.7387(6)	0.5696(4)	0.0720(13)
H11A	0.5281	0.6490	0.5930	0.086*
C12	0.5915(7)	0.8728(7)	0.6264(4)	0.0758(14)
H12A	0.5388	0.8709	0.6868	0.091*
C13	0.6741(6)	1.0079(6)	0.5957(4)	0.0689(13)
H13A	0.6790	1.0975	0.6339	0.083*
C14	0.7496(5)	1.0042(5)	0.5057(3)	0.0513(10)

intermolecular N(2)–H(2A)···I(2)#1 (symmetry code #1: $x - 1, y, z$) and N(3)–H(3B)···I(2)#1 hydrogen bonds make the complex forming a infinite chain. Neighboring chains are connected through N(5)–H(5A)···I(1)#2 (symmetry code #2: $-x + 2, -y + 2, -z + 1$) and N(6)–H(6B)···I(1)#3 (symmetry code #3: $x, y + 1, z$) hydrogen bonds, further stabilizing the 2-D structure.

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr. C* **71** (2015) 3–8.
3. Brandenburg, K.: DIAMOND. Visual crystal structure information system. Version 3.2i. Crystal Impact, Bonn, Germany (2012).
4. Zhao, Y.; Li, S.-H.; Zhang, L.: Crystal structure of poly [μ -2,6-dimethyl-pyridine-3,5-dicarboxylate- $\kappa^6 O, O', O'', O''', O''''$] calcium(II), C₉H₁₁CaNO₆. *Z. Kristallogr. NCS.* **230** (2015) 31–32.
5. Li, Y.-X.; Wen, D.-X.; An, M.-J.; Yang, H.-X.: Crystal structure of diaqua-(2,2'-(butane-1,4-diyl)-bis (5-carboxy-1H-imidazole-4-carboxylato)- $\kappa^4 O, O', N, N'$)cadmium(II)monohydrate, C₁₄H₁₈O₁₁N₄Cd. *Z. Kristallogr. NCS* **232** (2017) 545–547.
6. Zhao, L.; Wang, Z.-W.; Zhao, D.; Meng, X.-R.: Syntheses and crystal structures of three Cu(II) complexes based on 2,2'-(ethane-1,2-diyl)bis (1H-imidazole-4,5-dicarboxylic acid). *Synth. React. Inorg.* **44** (2014) 195–202.
7. Zhu, W.-F.; Zhu, X.: Crystal structure of poly[bis-m3-[(2-(1,2,4-triazol-1-yl)acetate- $\kappa^3 O:O':N$) cobalt(II)], [Co(C₄H₄N₃O₂)₂]_n, C₈H₈CoN₆O₄. *Z. Kristallogr. NCS* **229** (2014) 289–290.
8. Gu, W.; Miao, T.-T.; Hua, D.-W.; Jin, X.-Y.; Tao, X.-B.; Huang, C.-B.; Wang, S.-F.: Synthesis and *in vitro* cytotoxic evaluation of new 1H-benzo[d] imidazole derivatives of dehydroabietic acid. *Bioorg. Med. Chem. Lett.* **27** (2017) 1296–1300.
9. Kim, M. K.; Shin, H.; Park, K. S.; Kim, H.; Park, J.; Kim, K.; Nam, J.; Choo, H.; Chong, Y.: Benzimidazole derivatives as potent JAK1-selective inhibitors. *J. Med. Chem.* **58** (2015) 7596–7602.
10. Sharma, P.; Reddy, T. S.; Thummuri, D.; Senwar, K. R.; Kumar, N. P.; Naidu, V. G. M.; Bhargava, S. K.; Shankaraiah, N.: Synthesis and biological evaluation of new benzimidazole-thiazolidinedione hybrids as potential cytotoxic and apoptosis inducing agents. *Eur. J. Med. Chem.* **124** (2016) 608–621.
11. Zhu, H.-Z.; Geng, Z.; Zhang, S.; Liu, S.-C.: Preparation and structural characterization of [Cd(2-SC₅H₃NH-3-SiMe₃)₂]₂. *J. Struct. Chem.* **12** (1993) 72.
12. Zhao, W.-X.; Wen, P.-H.; Wang, Y.; Jiang, L.; Feng, G.-D.; Li, Z.-X.: Synthesis, Crystal structure and physical properties of a novel cadmium(II) supramolecular 3D framework. *Chinese J. Struct. Chem.* **34** (2015) 786–793.
13. Huang, Q.-Y.; Tang, W.-P.; Liu, C.-L.; Su, X.; Meng, X.-R.: Syntheses, structures, and properties of two 2-D Cd(II) complexes based on 2-(1H-imidazol-1-methyl)-1H-benzimidazole and polycarboxylate ligands. *J. Coord. Chem.* **67** (2014) 149–161.