Xia Wang, Ning Ling, Jing Yang and Ning Song*

Crystal structure of dichlorido bis[1-((2,4-dimethyl-1H-imidazol-1-yl)methyl)-1H-benzo[d][1,2,3]triazole- κN]cadmium(II), $\text{Cd}(\text{C}_{12}\text{H}_{13}\text{N}_5)_2\text{Cl}_2$

<https://doi.org/10.1515/ncrs-2018-0370>

Received September 14, 2018; accepted November 18, 2018;
available online December 15, 2018

Abstract

$[\text{Cd}(\text{C}_{12}\text{H}_{13}\text{N}_5)_2\text{Cl}_2]$, monoclinic, *Cc* (no. 9), $a = 9.3081(2)$ Å, $b = 11.7526(3)$ Å, $c = 24.9876(6)$ Å, $\beta = 94.184(2)^\circ$, $Z = 4$, $V = 2726.23(11)$ Å³, $R_{\text{gt}}(F) = 0.0412$, $wR_{\text{ref}}(F^2) = 0.0581$, $T = 293(2)$ K.

CCDC no.: 1879647

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All starting materials are commercially available, and are used without further purification. 1-[(2,4-Dimethyl-1H-imidazol-1-yl)methyl]-1H-benzotriazole (dmimb) was prepared according to the literature method [3]. The ligand dmimb (0.04 mmol, 0.009 g) in methanol (6 mL) was added dropwise to a methanol solution (6 mL) of CdCl_2 (0.04 mmol, 0.007 g) in methanol. The resulting solution was allowed

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	$0.16 \times 0.14 \times 0.13$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.03 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	29.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12268, 5833, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4973
$N(\text{param})_{\text{refined}}$:	338
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

to stand at room temperature. After two weeks colourless crystals were obtained.

Experimental details

H atoms were generated geometrically and refined as riding atoms with $\text{C}-\text{H} = 0.93$ Å and $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ for aromatic H atoms, with $\text{C}-\text{H} = 0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ for methylene H atoms, and with $\text{C}-\text{H} = 0.96$ Å and $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{C})$ for methyl H atoms.

Comment

In the past decades, the design and synthesis of coordination complexes have received rather considerable attention in the field of material chemistry and crystal engineering [3, 4]. Imidazole and benzotriazole derivatives have been widely used in the construction of complexes since they possess a wide variety of biological activity [5–7]. However, complexes with asymmetric imidazole and benzotriazole ligands are rare

*Corresponding author: Ning Song, College of Pharmacy, Henan University of Traditional Chinese Medicine, Zhengzhou 450046, P.R. China, e-mail: 646861300@qq.com

Xia Wang, Ning Ling and Jing Yang: College of Pharmacy, Henan University of Traditional Chinese Medicine, Zhengzhou 450046, P.R. China

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	1.0216(8)	0.1928(6)	0.0407(4)	0.050(2)
H1	1.0795	0.1979	0.0725	0.060*
C2	1.0485(8)	0.2561(5)	−0.0041(3)	0.064(2)
H2	1.1273	0.3050	−0.0027	0.077*
C3	0.9584(10)	0.2480(7)	−0.0518(3)	0.065(2)
H3	0.9793	0.2923	−0.0810	0.078*
C4	0.8435(10)	0.1785(6)	−0.0565(3)	0.059(2)
H4	0.7854	0.1740	−0.0883	0.070*
C5	0.8147(8)	0.1132(6)	−0.0118(3)	0.0442(19)
C6	0.9030(6)	0.1211(5)	0.0352(3)	0.0372(14)
C7	0.8866(7)	0.0177(5)	0.1243(2)	0.0459(16)
H7A	0.9904	0.0235	0.1304	0.055*
H7B	0.8595	−0.0603	0.1313	0.055*
C8	0.6865(6)	0.0791(5)	0.1802(2)	0.0364(14)
C9	0.8782(6)	0.1885(5)	0.1841(2)	0.0404(14)
H9	0.9688	0.2178	0.1786	0.049*
C10	0.7816(6)	0.2335(5)	0.2162(2)	0.0409(14)
C11	0.7935(7)	0.3357(6)	0.2518(3)	0.069(2)
H11A	0.7273	0.3931	0.2381	0.104*
H11B	0.8900	0.3650	0.2529	0.104*
H11C	0.7709	0.3148	0.2874	0.104*
C12	0.5894(7)	−0.0191(5)	0.1667(3)	0.062(2)
H12A	0.4931	−0.0002	0.1748	0.093*
H12B	0.6219	−0.0842	0.1874	0.093*
H12C	0.5908	−0.0362	0.1292	0.093*
C13	0.2069(8)	0.0627(6)	0.5164(3)	0.059(2)
H13	0.2721	0.0077	0.5068	0.071*
C14	0.2103(9)	0.1081(7)	0.5677(3)	0.077(2)
H14	0.2788	0.0817	0.5937	0.092*
C15	0.1146(10)	0.1916(7)	0.5814(3)	0.076(2)
H15	0.1213	0.2190	0.6164	0.091*
C16	0.0124(11)	0.2348(9)	0.5464(4)	0.075(3)
H16	−0.0518	0.2902	0.5564	0.089*
C17	0.0077(8)	0.1918(6)	0.4938(3)	0.0605(19)
C18	0.0993(9)	0.1052(7)	0.4802(3)	0.049(2)
C19	0.1122(8)	−0.0038(7)	0.3929(3)	0.067(2)
H19A	0.0369	−0.0228	0.3655	0.080*
H19B	0.1340	−0.0718	0.4140	0.080*
C20	0.2482(7)	0.1017(5)	0.3250(2)	0.0379(15)
C21	0.3795(8)	−0.0113(5)	0.3795(3)	0.0549(17)
H21	0.4065	−0.0633	0.4064	0.066*
C22	0.4666(8)	0.0377(5)	0.3453(2)	0.0480(17)
C23	0.6258(7)	0.0284(7)	0.3417(3)	0.083(3)
H23A	0.6450	−0.0122	0.3097	0.124*
H23B	0.6676	−0.0117	0.3724	0.124*
H23C	0.6668	0.1033	0.3406	0.124*
C24	0.1253(7)	0.1655(6)	0.2993(3)	0.0598(19)
H24A	0.1581	0.2130	0.2714	0.090*
H24B	0.0833	0.2121	0.3257	0.090*
H24C	0.0544	0.1131	0.2842	0.090*
Cd1	0.44408(4)	0.20826(3)	0.24023(2)	0.03632(10)
Cl1	0.41532(18)	0.40578(13)	0.26571(8)	0.0657(5)
Cl2	0.27411(17)	0.16683(17)	0.16269(7)	0.0658(5)
N1	0.7087(7)	0.0346(6)	−0.0048(3)	0.0641(18)
N2	0.7269(6)	−0.0059(4)	0.0434(2)	0.0563(14)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
N3	0.8447(6)	0.0453(4)	0.0690(2)	0.0413(14)
N4	0.8189(4)	0.0931(4)	0.16136(18)	0.0344(11)
N5	0.6604(5)	0.1631(4)	0.21298(18)	0.0353(11)
N6	−0.0846(9)	0.2176(7)	0.4497(4)	0.086(3)
N7	−0.0519(7)	0.1519(7)	0.4100(3)	0.082(2)
N8	0.0594(8)	0.0824(6)	0.4273(3)	0.0610(19)
N9	0.2417(6)	0.0308(4)	0.3668(2)	0.0488(13)
N10	0.3820(5)	0.1087(4)	0.31103(18)	0.0372(11)

[8, 9]. On the other hand, Cd(II)-connected coordination polymers have attracted considerable interest owing to its various coordination modes [10]. We are engaged in the synthesis of unsymmetrical N-heterocyclic ligands.

The title compound contains a mononuclear complex. As is shown in the figure, the Cd(II) atom is four-coordinated by two N atoms from two crystallographically independent dmimb ligands and two chlorido ligands in a distorted tetrahedral coordination (with Cd–N bond lengths of 2.232(4) and 2.236(4) Å, and a Cd–Cl bond lengths of 2.4270(16) and 2.4592(16) Å). The bond angles around Cd(II) range from 104.7 to 115.2°. In addition, the benzotriazole rings of adjacent molecules are parallel, with an interplanar distance of 3.536 Å. Thus π–π interactions are not to be ruled out. Bond lengths and angles are in accord with related structures [11].

Acknowledgements: This work was financially supported by the Science and Technology Department of Henan Province (no. 182102310315) and the Foundation for Henan Provincial Institute of Henan University of Chinese Medicine (2014KYYWF-QN11).

References

- Agilent Technologies. CrysAlis^{PRO} Software system, version 1.171.37.35. Agilent Technologies UK Ltd, Oxford, UK (2014).
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
- Moulton, B.; Zaworotko, M.-J.: From molecules to crystal engineering: supramolecular isomerism and polymorphism in network solids. *Chem. Rev.* **101** (2001) 1629–1658.
- Kitagawa, S.; Kitaura, R.; Noro, S.: Functional porous coordination polymers. *Angew. Chem. Int. Ed.* **43** (2004) 2334–2375.
- Luo, Y.; Zhang, S.; Liu, Z.-J.; Chen, W.; Fu, J.; Zeng, Q.-F.; Zhu, H.-L.: Synthesis and antimicrobial evaluation of a novel class of 1,3,4-thiadiazole: derivatives bearing 1,2,4-triazolo[1,5-*a*] pyrimidine moiety. *Eur. J. Med. Chem.* **64** (2013) 54–61.
- Fan, L.-Y.; Li, L.-K.; Xu, B.-J.; Qiao, M.; Li, J.-J.; Hou, H.-W.: Syntheses, crystal structures and luminescent properties of

- three complexes with mercapto-thiadiazole ligand involving in situ ligand synthesis. *Inorg. Chim. Acta* **423** (2014) 46–51.
7. Dubey, A.; Srivastava, S.-K.; Srivastava, S.-D.: Conventional and microwave assisted synthesis of 2-oxo-4-substituted aryl-azetidine derivatives of benzotriazole: a new class of biological compounds. *Bioorg. Med. Chem. Lett.* **21** (2011) 569–C573.
 8. Meng, X.-R.; Jin, S.-Z.; Hou, H.-W.; Du, C.-X.; Ng, S. W.: Self-assembly of four supramolecular architectures: from 0-D to 2-D frameworks based on an unsymmetrical *N*-heterocyclic ligand. *Inorg. Chim. Acta* **362** (2009) 1519–1527.
 9. Wang, X.; Shi, J.-J.; Zhang, S.-L.; Li, H.-B.: Crystal structure of dibromido-bis[1-[(2-methyl-1*H*-benzimidazol-1-yl)methyl]-1*H*-benzotriazole- κN]mercury(II), $\text{C}_{30}\text{H}_{26}\text{Br}_2\text{HgN}_{10}$. *Z. Kristallogr. NCS* **232** (2017) 79–80.
 10. Liang, Y.; Liu, M.; Zhang, N.; Wang, Q.-L.; Niu, Y.-Y.; Liu, X.-C.; Hou, H.-W.: Synthesis and structure of a CdI_2 coordination polymer with 1,1-(1,4-butanediyl)bis-1*H*-benzotriazole (bbbt). *J. Chem. Crystallogr.* **41** (2011) 617–620.
 11. Song, L.-Q.; Zhao, M.-H.; Cui, G.-S.; Gao, J.-H.; Lin, L.: Dichlorido-bis (1-[(2-methylbenzimidazol-1-yl- κN^3)methyl]benzotriazole)zinc. *Acta Crystallogr.* **E68** (2012) m1179.