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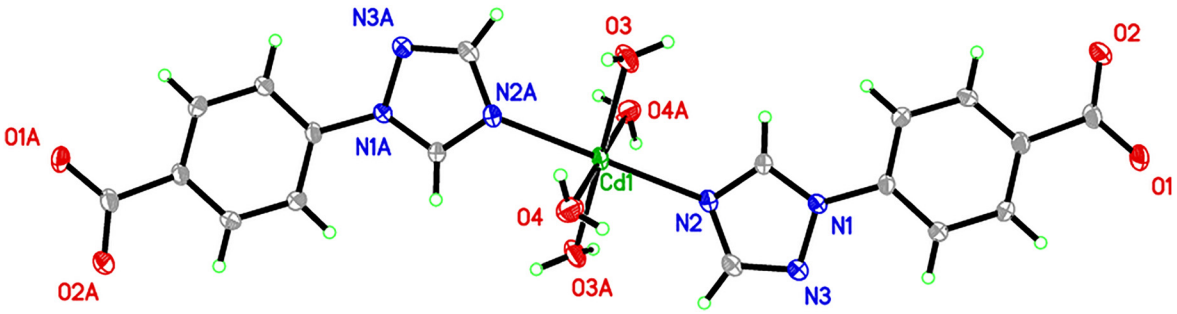
Crystal structure of tetraaqua-bis[4-(1*H*-1,2,4-triazol-1-yl)benzoato-k^1*N*]cadmium(II), $C_{18}H_{20}CdN_6O_8$



https://doi.org/10.1515/ncrs-2022-0205
Received April 21, 2022; accepted June 28, 2022;
published online July 13, 2022

Abstract

$C_{18}H_{20}CdN_6O_8$, triclinic, $P\bar{1}$ (no. 2), a = 6.1132(7) Å, b = 7.1270(9) Å, c = 12.3110(13) Å, α = 80.7010(10)°, β = 88.137(2)°, γ = 73.6530(10)°, V = 507.88(10) Å^3, Z = 1, $R_{gt}(F)$ = 0.0285, w$R_{ref}(F^2)$ = 0.0744, T = 298(2) K.

CCDC no.: 2130106

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:	Colourless block
Size:	0.49 × 0.40 × 0.39 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.14 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Advance, φ and ω
θ_{max}, completeness:	25.3°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2665, 1836, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1800
$N(param)_{refined}$:	168
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All the chemical reagents were purchased and used without further purification. $Cd(NO_3)_2 \cdot 4H_2O$ (0.0308 g, 0.1 mmol), 1-(4-carboxyphenyl)-1,2,4-triazole) ligand (Hcpt, 0.0189 g, 0.1 mmol) and DMF/H_2O (6 mL, 3:3, v/v) were placed in a 10 mL capped vial and used ultrasonic technique to get a clear solution. The vial was transferred to an 85 °C oven, heated for three days and cooled to room temperature at a rate of 10 °C/h. Colourless block crystals of the title complex were obtained, yielding 53% (based on the ligand).

Experimental details

The structure was solved by intrinsic phasing with the SHELXT program and refined with SHELXL program via OLEX2 software. The hydrogen atoms were placed in their idealized positions with isotropic thermal parameters.

Comment

As a polydentate organic ligand, 1-(4-carboxyphenyl)-1,2,4-triazole (Hcpt), which bearing both O and N coordinated sites, has been applied to construct metal complexes

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.500000	0.500000	0.500000	0.03121 (15)
O1	0.3459 (4)	0.9730 (3)	−0.37289 (17)	0.0437 (5)
O2	0.0123 (4)	1.0665 (4)	−0.29116 (18)	0.0496 (6)
O3	0.1834 (4)	0.7694 (4)	0.4822 (2)	0.0459 (6)
O4	0.7043 (4)	0.6973 (4)	0.5618 (2)	0.0429 (5)
N1	0.5912 (4)	0.6459 (3)	0.14238 (18)	0.0274 (5)
N2	0.6134 (4)	0.5599 (4)	0.31996 (19)	0.0326 (5)
N3	0.8183 (4)	0.5642 (4)	0.1655 (2)	0.0362 (6)
C1	0.2220 (5)	0.9834 (4)	−0.2892 (2)	0.0317 (6)
C2	0.3307 (4)	0.8891 (4)	−0.1777 (2)	0.0267 (5)
C3	0.5641 (5)	0.8233 (4)	−0.1588 (2)	0.0340 (6)
H3	0.662761	0.834787	−0.217016	0.041*
C4	0.6526 (5)	0.7403 (4)	−0.0537 (2)	0.0335 (6)
H4	0.809457	0.696503	−0.041612	0.040*
C5	0.5056 (4)	0.7233 (4)	0.0327 (2)	0.0252 (5)
C6	0.2721 (5)	0.7801 (5)	0.0140 (2)	0.0387 (7)
H6	0.173452	0.761362	0.071238	0.046*
C7	0.1884 (5)	0.8641 (5)	−0.0899 (2)	0.0370 (7)
H7	0.031427	0.905458	−0.101821	0.044*
C8	0.8216 (5)	0.5147 (5)	0.2731 (2)	0.0360 (6)
H8	0.955519	0.453876	0.313707	0.043*
C9	0.4737 (5)	0.6430 (4)	0.2351 (2)	0.0327 (6)
H9	0.316324	0.692255	0.239585	0.039*
H3A	0.119 (8)	0.824 (7)	0.420 (4)	0.072 (13)*
H3B	0.211 (8)	0.860 (7)	0.521 (4)	0.070 (14)*
H4A	0.760 (10)	0.754 (9)	0.513 (5)	0.10 (2)*
H4B	0.603 (9)	0.783 (8)	0.585 (4)	0.078 (16)*

[5–10]. However, it should be noted that most of the published Cd(II)-based complexes are complicated structures. To further enrich the family of Hcpt-based metal complexes, the title complex was synthesized under hydrothermal condition.

The complex crystallizes in the triclinic system with a space group of $P\bar{1}$ (no. 2). The asymmetric unit consists of one deprotonated Hcpt ligand, one half of Cd(II) ion and two coordinated water molecules. The Cd(II) ion is six-coordinated with two triazolyl nitrogen atoms (N2, N2$^{1-x,\ 1-y,\ 1-z}$) derived from two different deprotonated Hcpt ligands and four oxygen atoms (O3, O3$^{1-x,\ 1-y,\ 1-z}$, O4, O4$^{1-x,\ 1-y,\ 1-z}$) originated from four coordinated water molecules. The Cd–N and Cd–O bond lengths are in the range of 2.298(2)–2.348(2) Å, which are comparable to the other Cd(II) complexes [11, 12]. The complex is further linked by the coordinated water molecules and carboxylate groups via the classical O–H—O hydrogen bond to form a three-dimensional (3D) network. Apart from the hydrogen bonding, the π-π stacking, distance range: 3.7598(17)–3.9104(17) Å, don't play an important role in the process of forming the network.

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

Research funding: This work was supported by the Research Fund for the Doctoral Program of Weifang University (NO. 2021BS09).

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

References

1. Bruker. *SAINT and SADABS*; Bruker AXS Inc: Madison, Wisconsin, USA, 2000.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
5. Singh M., Senthilkumar S., Rajput S., Neogi S. Pore-functionalized and hydrolytically robust Cd(II)-metal–organic framework for highly selective, multicyclic CO_2 adsorption and fast-responsive luminescent monitoring of Fe(III) and Cr(VI) ions with notable sensitivity and reusability. *Inorg. Chem.* 2020, *59*, 3012–3025.
6. Li T., Liu X., Huang Z.-P., Lin Q., Lin C.-L., Zhan Q.-G., Xu X.-D., Cai Y.-P. Metal cation-dependent construction of two 3D interpenetrating networks based on the ligand 1-(4-carboxyphenyl)-1,2,4-triazole. *Inorg. Chem. Commun.* 2014, *39*, 70–74.
7. Li J., Zhang G., Li Y.-T., Wang X., Zhu J.-Q., Tian Y.-Q. Metal–organic frameworks derived from divalent metals and 4-(1*H*-1, 2, 4-triazol-1-yl)benzoic acid. *Open J. Inorg. Chem.* 2012, *2*, 58–66.
8. Zhu A.-X., Qiu Z.-Z., Yang L.-B., Fang X.-D., Chen S.-J., Xu Q.-Q., Li Q.-X. A luminescent cadmium(II) metal–organic framework based on a triazolate-carboxylate ligand exhibiting selective gas adsorption and guest-dependent photoluminescence properties. *CrystEngComm* 2015, *17*, 4787–4792.
9. Tian H.-R., Zhang Z., Dang T.-Y., Liu S.-M., Lu Y., Liu S.-X. Hollow lindqvist-like-shaped {V_6} cluster-based metal–organic framework for the highly efficient detoxification of mustard gas simulant. *Inorg. Chem.* 2021, *60*, 840–845.
10. Du M., Li C.-P., Chen M., Ge Z.-W., Wang X., Wang L., Liu C.-S. Divergent kinetic and thermodynamic hydration of a porous Cu(II) coordination polymer with exclusive CO_2 sorption selectivity. *J. Am. Chem. Soc.* 2014, *136*, 10906–10909.
11. Ou Y.-C., Chen H.-C., Liang Y.-H., Tang J.-J., Feng S.-B., Peng L., Wu J.-Z. Crystal structures, *in situ* synthesis in aqua conditions and luminescent properties of benzimidazole ligands and cadmium coordination complexes. *Inorg. Chim. Acta* 2015, *435*, 215–222.
12. Wang D.-W., Wang T., Yan T., Du L., Zhao Q.-H. Crystal structures and spectroscopic characterizations of two Cd(II) complexes based on [1,2,4]-triazole derivatives. *Chin. J. Inorg. Chem.* 2017, *33*, 1443–1449.