Crystal structure of bisnitrate-bis(1-(4-((1*H*-imidazol-1yl)methyl)-benzyl)-1*H*-imidazole)cadmium(II), $[Cd(NO_3)_2(C_{14}H_{14}N_4)_2]_n$

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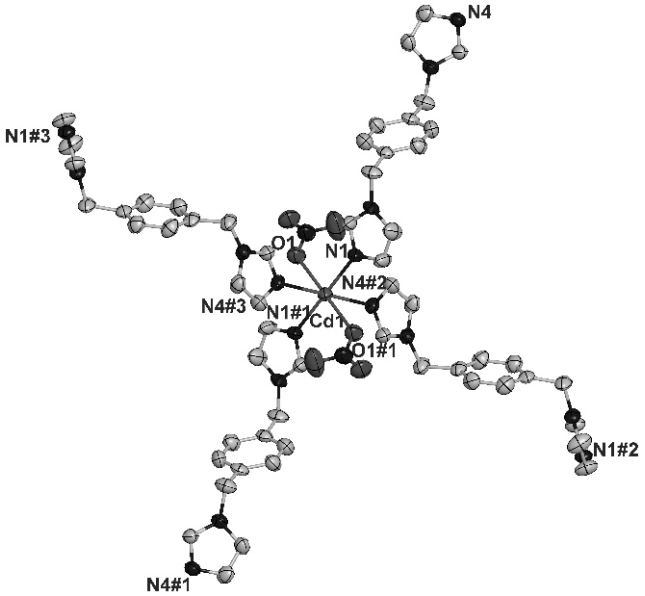

Abstract

$C_{28}H_{28}CdN_{10}O_6$, monoclinic, $P12_1/c1$ (no. 14), a = 8.621(2) Å, b = 20.782(4) Å, c = 8.667(2) Å, β = 102.24(3)°, V = 1517.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.068, $wR_{ref}(F^2)$ = 0.183, T = 296 K.

Source of material

All chemicals and solvents were of reagent grade and were used in the synthesis without further purification. An aqueous solution of $Cd(NO_3)_2 \cdot 4H_2O$ (3 ml, 15.42 mg, 0.05 mmol) was slowly added dropwise to a solution of 1-(4-((1*H*-imidazol-1-yl)methyl)benzyl)-1*H*-imidazole (bix, 24 mg, 0.1 mmol) in CH_3OH (3 ml) and the mixture was stirred for 30 min. The resultant colorless solution was allowed to stand in the darkness at room temperature for a week to give colorless needle-shaped crystals which were collected, washed with deionized water and dried in air. Elemental analysis — found: C, 47.17 %; H, 3.96 %; N, 19.65 %; calculated for $CdC_{28}H_{28}N_{10}O_6$: C, 47.20 %; H, 3.95 %; N, 19.59 %.

Experimental details

Hydrogen atoms on carbon atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

The connection of metal-organic coordination polymers based on complexes of transition metals and multifunctional bridging ligands has proven to be a promising field due to the intriguing network topologies and potential functions as new classes of materials [1-3]. Over the past two decades, different classes of coordination polymers have been successfully designed and synthesized. Recently, bridging ligands containing N or/and O, for example multidentate aromatic polycarboxylate [4-8], have been widely used for syntheses of coordination polymers of transition metals [9-10].

In the title crystal structure, there are one cadmium ion, two nitrate ions and two 1-(4-((1*H*-imidazol-1-yl)methyl)benzyl)-1*H*-imidazole molecules per asymmetric unit. The center Cd(II) is six-coordinated by four N atoms (N1,N1#1,N4,N4#1) from four bridging bidentate ligand molecules 1-(4-((1*H*-imidazol-1-yl)methyl)benzyl)-1*H*-imidazole and two O atoms (O1,O1#1) from two nitrate ions forming an octahedron. The bond lengths are d(Cd1—N1) = 2.2887(53) Å, d(Cd1—N4) = 2.3857(53) Å, and d(Cd1—O1) = 2.3882(56) Å. Since Cd(II) is located at a symmetry centre, there are three near 180° bond angles (∠N1–Cd–N1#1 = 180.0(2)°, ∠N4#2–Cd–N4#3 = 180.0(2)° and ∠O1–Cd–O1#1 = 180.0°. Symmetry codes #1:1–x,1–y,–z; #2: 1+x,3/2–y,–1/2+z; #3: –x,–1/2+y,1/2–z.), but 90° bond angles have not been observed (∠N1#1–Cd1–N4 = 91.0(2)°, ∠N1–Cd1–N4#2 = 89.0(2)°, ∠N1#1–Cd1–N4#3 = 89.0(2)°, ∠N1–Cd1–N4#3 = 91.0(2)°, ∠N1#1–Cd1–O1 = 84.5(2)°, ∠N1–Cd1–O1 = 95.6(2)°, ∠N4#2–Cd1–O1 = 100.8(2)°, ∠N4#3–Cd1–O1 = 79.2(2)°. Symmetry codes #1:1–x,1–y,–z; #2: 1+x,3/2–y,–1/2+z; #3: –x,–1/2+y, 1/2–z). So, Cd(II) has a slightly distorted octahedral environment. In addition, other N-donors of the four bidentate ligands bridge two adjacent Cd(II) ions. Through the bridging ligand, Cd(II) ions are united together to form a new 2D coordination polymer.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.19 × 0.20 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.79 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13650, 2670
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2216
$N(param)_{refined}$:	199
Programs:	SHELXS-97, SHELXL-97, SHELXTL [11], DIAMOND [12]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.2279	0.5322	0.2214	0.060
H(2)	4*e*	0.6749	0.5778	0.3445	0.073
H(3)	4*e*	0.5452	0.6116	0.5551	0.083
H(4A)	4*e*	0.2629	0.5939	0.6167	0.065
H(4B)	4*e*	0.1502	0.5479	0.5003	0.065
H(6)	4*e*	–0.0449	0.5922	0.2839	0.067
H(7)	4*e*	–0.1925	0.6783	0.1710	0.066
H(9)	4*e*	0.0838	0.7966	0.4729	0.062
H(10)	4*e*	0.2340	0.7102	0.5793	0.066
H(11A)	4*e*	–0.0911	0.8388	0.2223	0.068
H(11B)	4*e*	–0.2161	0.7920	0.1230	0.068
H(12)	4*e*	–0.4457	0.7541	0.3075	0.066
H(13)	4*e*	–0.5958	0.8295	0.4323	0.070
H(14)	4*e*	–0.2161	0.9179	0.3686	0.056

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	2*d*	½	½	0	0.0427(5)	0.0410(4)	0.0448(4)	–0.0067(3)	0.0193(3)	–0.0063(3)
O(1)	4*e*	0.2266(7)	0.4985(2)	–0.1304(7)	0.046(3)	0.064(3)	0.084(3)	0.008(2)	0.007(2)	–0.015(2)
O(2)	4*e*	–0.0016(6)	0.5356(3)	–0.2455(8)	0.041(3)	0.094(4)	0.111(5)	0.006(3)	0.004(3)	–0.012(4)
O(3)	4*e*	0.1607(9)	0.5916(3)	–0.080(1)	0.119(6)	0.073(5)	0.170(8)	0.013(4)	–0.020(6)	–0.036(5)
N(1)	4*e*	0.4588(6)	0.5448(2)	0.2292(6)	0.042(3)	0.047(3)	0.046(3)	–0.002(2)	0.019(3)	–0.008(2)
N(2)	4*e*	0.3423(6)	0.5750(2)	0.4190(6)	0.050(3)	0.045(3)	0.035(3)	0.007(2)	0.018(2)	–0.004(2)
N(3)	4*e*	–0.2841(6)	0.8274(2)	0.3122(6)	0.047(3)	0.040(3)	0.045(3)	0.007(2)	0.014(3)	0.000(2)
N(4)	4*e*	–0.4221(6)	0.8968(2)	0.4226(6)	0.046(3)	0.037(3)	0.049(3)	0.008(2)	0.015(3)	0.000(2)
N(5)	4*e*	0.1263(8)	0.5401(3)	–0.1465(8)	0.046(3)	0.064(3)	0.084(3)	0.008(2)	0.007(2)	–0.015(2)
C(1)	4*e*	0.3242(8)	0.5476(3)	0.2787(8)	0.039(4)	0.059(4)	0.054(4)	–0.010(3)	0.015(3)	–0.014(3)
C(2)	4*e*	0.5680(8)	0.5725(4)	0.3451(9)	0.034(4)	0.085(5)	0.064(5)	–0.007(4)	0.014(4)	–0.020(4)
C(3)	4*e*	0.4966(8)	0.5912(5)	0.4619(9)	0.047(5)	0.097(7)	0.059(5)	–0.001(4)	0.002(4)	–0.032(4)
C(4)	4*e*	0.2155(9)	0.5861(3)	0.5064(8)	0.058(4)	0.063(5)	0.050(4)	0.015(3)	0.032(3)	0.008(3)
C(5)	4*e*	0.1120(8)	0.6428(3)	0.4418(7)	0.045(4)	0.046(4)	0.044(4)	0.006(3)	0.025(3)	0.001(3)
C(6)	4*e*	–0.0174(9)	0.6335(3)	0.3208(9)	0.057(5)	0.048(4)	0.066(5)	0.003(3)	0.018(4)	–0.008(3)
C(7)	4*e*	–0.1063(9)	0.6851(3)	0.2542(9)	0.052(4)	0.055(4)	0.058(4)	0.006(3)	0.008(3)	–0.015(4)
C(8)	4*e*	–0.0707(8)	0.7470(3)	0.3079(7)	0.049(4)	0.052(4)	0.041(3)	0.011(3)	0.023(3)	–0.004(3)
C(9)	4*e*	0.0580(8)	0.7556(3)	0.4325(9)	0.054(4)	0.041(4)	0.062(4)	–0.005(3)	0.015(4)	–0.012(3)
C(10)	4*e*	0.1473(9)	0.7037(3)	0.4965(8)	0.054(4)	0.059(5)	0.050(4)	0.002(3)	0.008(3)	–0.011(3)
C(11)	4*e*	–0.1642(9)	0.8040(3)	0.2296(9)	0.061(5)	0.061(4)	0.055(4)	0.020(4)	0.026(4)	0.005(4)
C(12)	4*e*	–0.4175(8)	0.7965(3)	0.3346(9)	0.051(4)	0.037(4)	0.074(5)	0.002(3)	0.008(4)	–0.006(3)
C(13)	4*e*	–0.5004(9)	0.8384(3)	0.403(1)	0.052(4)	0.045(4)	0.080(5)	–0.002(3)	0.020(4)	0.010(4)
C(14)	4*e*	–0.2935(8)	0.8867(3)	0.3682(8)	0.043(4)	0.042(4)	0.058(4)	0.002(3)	0.020(3)	0.002(3)

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References

1. Min, K. S.; Suh, M. P.: Silver(I)-Polynitrile Network Solids for Anion Exchange: Anion-Induced Transformation of Supramolecular Structure in the Crystalline State. J. Am. Chem. Soc. **122** (2000) 6834-6840.
2. Eddaoudi, M.; Moler, D. B.; Li, H.; Chen, B.; Reineke,T. M.; O'Keeffe, M.; Yaghi, O. M.: Modular Chemistry: Secondary Building Units as a Basis for the Design of Highly Porous and Robust Metal-Organic Carboxylate Frameworks. Acc. Chem. Res. **34** (2001) 319-330.
3. Moulton, B.; Zaworotko, M. J.: From Molecules to Crystal Engineering: Supramolecular Isomerism and Polymorphism in Network Solids. Chem. Rev. **101** (2001) 1629-1658.
4. Tao, J.; Tong, M.-L.; Shi, J.-X.; Chen, X.-M.; Ng, S. W.: Blue photoluminescent zinc coordination polymers with supertetranuclear cores. Chem.Commun. **20** (2000) 2043-2044.
5. Yaghi, O. M.; Li, H.: T-Shaped Molecular Building Units in the Porous Structure of $Ag(4,4'\text{-bpy}) \cdot NO_3$. J. Am. Chem. Soc. **118** (1996) 295-296.
6. Barthelte, K.; Riou, D.; Ferey, G.: $[V^{III}(H_2O)]_3O(O_2CC_6H_4CO_2)_3 \cdot$ (Cl, $9H_2O$) (MIL-59): a rare example of vanadocarboxylate with a magnetically frustrated three-dimensional hybrid framework. Chem. Commun. **14** (2002) 1492- 1493.
7. Hong, C. S.; Do. Y.: Hydrogen- and Covalent-Bond-Linked 3D Manganese Array. Inorg. Chem. **37** (1998) 4470-4472.
8. Li, H.; Davis, C. E.; Groy, T. L.; Kelley, D. G.; Yaghi, O. M.: Coordinatively Unsaturated Metal Centers in the Extended Porous Framework of $Zn_3(BDC)_3 \cdot 6CH_3OH$ (BDC=1,4-Benzenedicarboxylate). J. Am. Chem. Soc. **120** (1998) 2186-2187.
9. Tao, J.; Zhang, Y.; Tong, M.-L.; Chen, X.-M.; Yuen, T.; Lin, C.-L.; Huang, X.-Y.; Li, J.: A mixed-valence copper coordination polymer generated by hydrothermal metal/ligand redox reactions. Chem. Commun. **13** (2002) 1342-1343.
10. Lin, W.-B.; Wang, Z.-Y.; Ma, L.: A Novel Octupolar Metal-Organic NLO Material Based on a Chiral 2D Coordination Network. J. Am. Chem. Soc. **121** (1999) 11249-11250.
11. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
12. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 2.0f. Crystal Impact, Bonn, Germany 1998.