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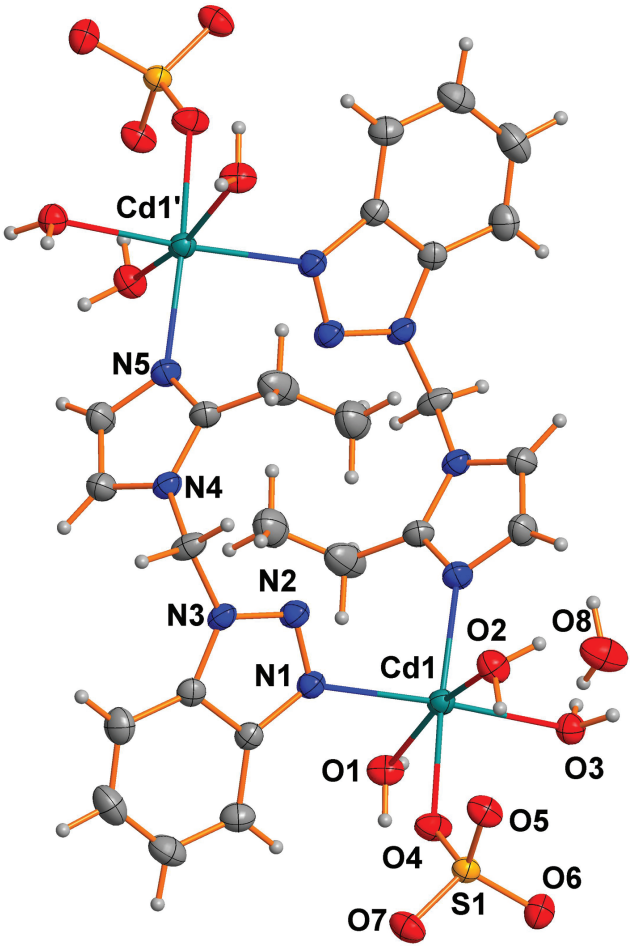
Crystal structure of *cyclo*[hexaqua-bis[1-[(2-ethyl-1*H*-imidazol-1-yl) methyl]-1*H*-benzotriazole-κ²*N:N'*]-bis(sulfato-κ¹*O*)dicadmium(II)] monohydrate, $C_{24}H_{42}Cd_2N_{10}O_{16}S_2$



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Abstract

{[$Cd_2(C_{12}H_{13}N_5)_2(H_2O)_6(SO_4)_2$]$(H_2O)_2$}, triclinic, $P\bar{1}$, a = 8.2977(5) Å, b = 10.2955(6) Å, c = 12.0979(9) Å, α = 103.879(6)°, β = 100.039(6)}°, γ = 104.567(5) V = 940.32(11) Å^3, Z = 1, $R_{gt}(F)$ = 0.0282, $wR_{ref}(F^2)$ = 0.0745, T = 291 K.

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The crystal structure of the title complex is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Prismatic, colorless
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.32 mm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω-scans
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7804, 3844, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3486
$N(param)_{refined}$:	269
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3]

Source of material

The compound 1-[(2-ethyl-1*H*-imidazol-1-yl)methyl]-1*H*-benzotriazole (0.04 mmol, 0.0096 g) in methanol (6 mL) was added dropwise to a methanol solution (6 mL) of $CdSO_4$ (0.04 mmol, 0.0083 g). The resulting solution was allowed to stand at room temperature. After two weeks colourless crystals of the title complex were obtained.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Compounds with *N*-donor, especially imidazole and benzotriazole derivatives, have been extensively studied, since they possess a wide variety of biological activity [4, 5]. However, compounds with asymmetric imidazole and benzotriazole ligands are rarely used in coordination chemistry [6–8]. We are engaged in the synthesis of unsymmetrical *N*-heterocyclic ligands and thus we synthesized compound 1-[(2-ethyl-1*H*-imidazol-1-yl)methyl]-1*H*-benzotriazole (bmei) [9].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.28437(2)	0.148428(19)	0.172881(16)	0.02552(8)
S1	0.27033(8)	−0.20732(7)	0.06087(6)	0.02438(15)
O1	−0.0051(2)	0.1054(2)	0.15300(19)	0.0338(5)
H1A	−0.0569	0.0178	0.1198	0.051*
O2	0.5534(3)	0.1271(2)	0.17842(19)	0.0354(5)
H2A	0.5464	0.0400	0.1493	0.053*
H2B	0.6035	0.1756	0.1379	0.053*
O3	0.2508(3)	0.1629(2)	−0.02268(18)	0.0310(4)
H3A	0.2158	0.2337	−0.0277	0.046*
O4	0.1997(3)	−0.0941(2)	0.1088(2)	0.0353(5)
O5	0.4602(2)	−0.1562(2)	0.10428(19)	0.0344(5)
O6	0.2216(3)	−0.2450(2)	−0.06870(18)	0.0382(5)
O7	0.2010(3)	−0.3294(2)	0.09930(19)	0.0369(5)
O8	0.1576(4)	0.3870(3)	−0.0555(3)	0.0522(7)
N1	0.3071(3)	0.1191(2)	0.3630(2)	0.0297(5)
N2	0.4348(3)	0.2102(2)	0.4462(2)	0.0314(5)
N3	0.4382(3)	0.1700(2)	0.54425(19)	0.0278(5)
N4	0.5527(3)	0.3861(2)	0.7037(2)	0.0271(5)
N5	0.6020(3)	0.6139(2)	0.7670(2)	0.0327(5)
C1	0.2600(4)	−0.0364(3)	0.5948(3)	0.0389(7)
H1	0.3188	−0.0153	0.6729	0.047*
C2	0.1185(4)	−0.1521(4)	0.5406(3)	0.0468(9)
H2	0.0789	−0.2105	0.5844	0.056*
C3	0.0302(4)	−0.1867(3)	0.4221(3)	0.0426(8)
H3	−0.0647	−0.2668	0.3900	0.051*
C4	0.0814(4)	−0.1047(3)	0.3529(3)	0.0344(7)
H4	0.0240	−0.1277	0.2742	0.041*
C5	0.2243(3)	0.0153(3)	0.4061(2)	0.0256(6)
C6	0.3093(3)	0.0478(3)	0.5234(2)	0.0264(6)
C7	0.5723(4)	0.2504(3)	0.6515(3)	0.0324(7)
H7A	0.5686	0.1973	0.7078	0.039*
H7B	0.6838	0.2644	0.6336	0.039*
C8	0.4360(4)	0.4073(3)	0.7661(3)	0.0357(7)
H8	0.3524	0.3383	0.7801	0.043*
C9	0.4637(4)	0.5471(3)	0.8042(3)	0.0367(7)
H9	0.4007	0.5914	0.8481	0.044*
C10	0.6543(4)	0.5139(3)	0.7067(3)	0.0314(6)
C11	0.8022(5)	0.5380(4)	0.6522(4)	0.0556(10)
H11A[a]	0.8738	0.6346	0.6895	0.067*
H11B[a]	0.8698	0.4786	0.6720	0.067*
H11C[b]	0.7668	0.4686	0.5754	0.067*
H11D[b]	0.8191	0.6291	0.6389	0.067*
C12[a]	0.7690(8)	0.5142(6)	0.5290(5)	0.0550(13)
H12A[a]	0.7004	0.4184	0.4897	0.082*
H12B[a]	0.8759	0.5321	0.5064	0.082*
H12C[a]	0.7083	0.5760	0.5075	0.082*
C12A[b]	0.9588(13)	0.5349(11)	0.7061(10)	0.0550(13)
H12D[b]	1.0031	0.6082	0.7797	0.082*
H12E[b]	1.0364	0.5489	0.6566	0.082*
H12F[b]	0.9476	0.4455	0.7200	0.082*
H1B	−0.056(4)	0.152(3)	0.122(3)	0.054(11)*
H3B	0.352(5)	0.165(4)	−0.051(4)	0.064(12)*
H8A	0.049(8)	0.364(5)	−0.065(5)	0.106(19)*
H8B	0.207(6)	0.474(5)	−0.007(4)	0.070(14)*

Occupancies: [a] = 0.645(5), [b] = 0.355(5)

The title compound is a dinuclear complex. As is shown in the figure, the Cd(II) atom is six-coordinated by two N atoms from two bridging bmei ligands, three O atoms from three water molecules, and one O atom from one sulfate anion in a distorted octahedral coordination. Around Cd1, the equatorial positions are occupied by O3, O4, N1 and N5A (the mean deviation from plane is 0.0740 Å), while O1, O2 are in axial positions. Notably, the methyl group of the organic ligand is disordered over two positions. The Cd(II) atoms are connected by the bmei ligands, leading to a dimer. The distance between two Cd atoms is 8.8 Å. In addition, the benzotriazole rings in adjacent complexes have an average interplanar distance of 3.462 Å. Thus, we suggest π−π interactions.

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References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.35.19, Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: A short history of *SHELX*. Acta Crystallogr. **A64** (2008) 112–122.
3. 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. **42** (2009) 339–341.
4. Luo, Y.; Zhang, S.; Liu, Z.-J.; Chen, W.; Fu, J.; Zeng, Q.-F.; Zhu, H.-L.: Synthesis and antimicrobical 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.
5. 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.
6. Hu, M.-C.; Wang, Y.; Zhai, Q.-G.; Li, S.-N.; Jiang, Y.-C.; Zhang, Y.: Synthesis, crystal structures, and photoluminescent properties of the Cu(I)/X/alpha,omega-bis(benzotriazole)alkane hybrid family (X = Cl, Br, I, and CN). Inorg. Chem. **48** (2009) 1449–1468.
7. 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. Chem. Acta **362** (2009) 1519–1527.
8. Wang, X.; Shi, J.-J.; Zhang, S.-L.; Li, H.-B.: Crystal structure of dibromido-bis[μ-1-[(2-methyl-1*H*-benzoimidazol-1-yl)methyl]-1*H*-benzotriazole-κ*N*]mercury(II), $C_{30}H_{26}Br_2HgN_{10}$. Z. Kristallogr. NCS **232** (2017) 79–80.
9. Wang, X.; Niu, J.-L.: Bis[μ_1[(2-ethyl-1*H*-imidazol-1-yl)methyl]-1*H*-Benzotriazole]bis[diiodinecadmium(II)]. Acta Crystallogr. **E67** (2011) m882.