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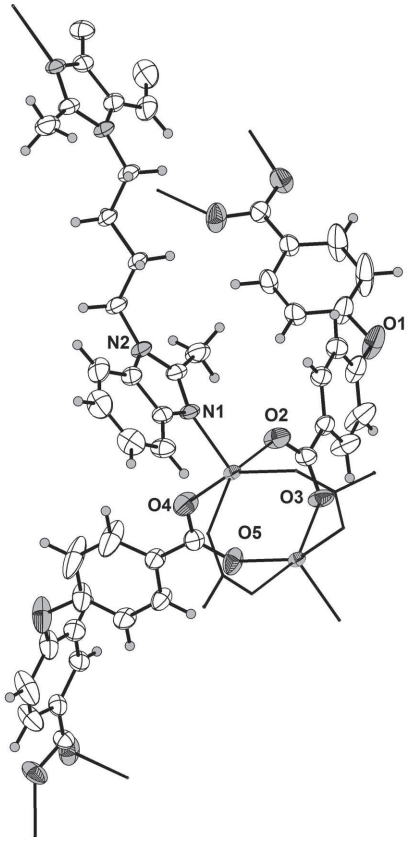
Crystal structure of poly[1,4-bis(2-methylbenzimidazol)butane-κ^2*N*:*N*′)bis(4,4′-oxybis(benzoato-κ^4*O*,*O*′:*O*″,*O*‴)dicadmium] monohydrate, $C_{48}H_{38}Cd_2N_4O_{10}$



DOI 10.1515/ncrs-2016-0003
Received January 3, 2016; accepted March 14, 2016; available online April 7, 2016

Abstract

$C_{48}H_{38}Cd_2N_4O_{10}$, monoclinic, $C2/c$ (no. 15), $a = 22.112(4)$ Å, $b = 9.2100(18)$ Å, $c = 22.977(5)$ Å, $\beta = 93.24(3)°$, $V = 4671.8(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0341$, $wR_{ref}(F^2) = 0.0868$, $T = 293(2)$ K.

CCDC no.: 1465345

A selected part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless, Blocks, Size 0.35 × 0.30 × 0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.7 cm^{-1}
Diffractometer, scan mode:	Rigaku Saturn 724 CCD, ω
$2\theta_{max}$, completeness:	55.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6969, 4347
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3990
$N(param)_{refined}$:	290
Programs:	SHELX [14], Bruker programs [15]

Source of material

The title compound was prepared under the hydrothermal conditions. A mixture of 1,4-bis(2-methylbenzimidazol) butane (63.6 mg, 0.2 mmol), $Cd(NO_3)_2{\cdot}4H_2O$ (30.8 mg, 0.1 mmol), 4,4′-oxybis(benzoic acid) (51.6 mg, 0.2 mmol), and H_2O (10 mL) were placed in a Teflon-lined stainless steel vessel, heated to 130°C for 2 day, and then cooled to room temperature for 24 h. Colorless block crystals of the title complex were obtained.

Experimental details

Absorption corrections were applied by using a multi-scan program [15]. The structures were solved by direct methods and refined with the SHELXL [14] crystallographic software package. The hydrogen atoms were placed at calculated positions and refined as riding atoms fixed with isotropic displacement parameters.

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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	0.054823(9)	0.38723(2)	1.008342(9)	0.03296(10)
N1	0.13010(12)	0.2282(3)	1.02595(12)	0.0413(6)
N2	0.20242(13)	0.0685(3)	1.01008(14)	0.0515(7)
O1	−0.17599(13)	−0.0664(4)	0.76137(15)	0.0999(13)
O2	−0.01252(12)	0.2631(3)	0.95376(13)	0.0686(8)
O3	−0.08803(13)	0.4199(3)	0.94004(13)	0.0691(8)
O4	0.08127(14)	0.5118(3)	0.92995(12)	0.0744(9)
O5	0.00245(13)	0.6579(4)	0.91753(13)	0.0766(9)
C1	0.22397(16)	0.1506(4)	1.05691(18)	0.0535(9)
C2	0.27826(18)	0.1497(5)	1.0900(2)	0.0715(13)
H2	0.3094	0.0860	1.0823	0.086*
C3	0.2837(2)	0.2483(6)	1.1352(2)	0.0837(15)
H3	0.3195	0.2508	1.1585	0.100*
C4	0.2377(2)	0.3433(5)	1.1470(2)	0.0753(13)
H4	0.2430	0.4067	1.1784	0.090*
C5	0.18433(18)	0.3463(4)	1.11338(17)	0.0571(10)
H5	0.1535	0.4106	1.1214	0.068*
C6	0.17828(16)	0.2502(3)	1.06731(15)	0.0455(8)
C7	0.14619(17)	0.1180(3)	0.99313(17)	0.0482(9)
C8	0.10778(19)	0.0515(5)	0.94570(19)	0.0681(11)
H8A	0.0903	−0.0367	0.9594	0.102*
H8B	0.1320	0.0302	0.9134	0.102*
H8C	0.0760	0.1177	0.9335	0.102*
C9	0.23644(18)	−0.0481(4)	0.98354(19)	0.0625(11)
H9A	0.2790	−0.0223	0.9853	0.075*
H9B	0.2230	−0.0564	0.9428	0.075*
C10	0.22939(17)	−0.1940(4)	1.01264(18)	0.0546(9)
H10A	0.2391	−0.1847	1.0542	0.066*
H10B	0.1877	−0.2259	1.0072	0.066*
C11	0.05375(18)	0.6158(4)	0.90615(16)	0.0494(9)
C12	0.08393(16)	0.6971(4)	0.85938(15)	0.0502(9)
C13	0.1400(2)	0.6615(7)	0.8431(3)	0.139(3)
H13	0.1593	0.5810	0.8600	0.167*
C14	0.05737(17)	0.8153(4)	0.83260(16)	0.0569(10)
H14	0.0188	0.8427	0.8424	0.068*
C15	−0.06185(16)	0.3034(4)	0.93018(15)	0.0466(8)
C16	−0.09257(15)	0.2021(4)	0.88659(15)	0.0465(8)
C17	−0.1370(2)	0.2523(5)	0.84737(19)	0.0763(14)
H17	−0.1497	0.3483	0.8496	0.092*
C18	−0.1630(2)	0.1628(6)	0.8048(2)	0.0937(18)
H18	−0.1918	0.1989	0.7774	0.112*
C19	−0.14607(17)	0.0217(5)	0.80330(18)	0.0695(13)
C20	−0.10366(18)	−0.0338(4)	0.84276(18)	0.0618(11)
H20	−0.0932	−0.1315	0.8417	0.074*
C21	−0.07643(16)	0.0579(4)	0.88456(16)	0.0503(8)
H21	−0.0471	0.0216	0.9113	0.060*
C22	−0.14178(18)	−0.1433(5)	0.7233(2)	0.0725(13)
C23	−0.1692(2)	−0.2596(8)	0.6977(3)	0.164(4)
H23	−0.2079	−0.2864	0.7076	0.196*
C24	−0.08605(18)	−0.1043(4)	0.70864(17)	0.0581(10)
H24	−0.0670	−0.0241	0.7260	0.070*

Discussion

The synthesis and structural characterization of metal-organic frameworks (MOFs) have been quickly developed because of their novel structures, intriguing topologies and potential applications [1–4]. In particular, the great mass of organic ligands containing coordination sites of N and/or O donors are wildly utilized as designing points in the construction of MOFs [5–7]. Among them, the flexible N-bridging ligands bearing alkyl spacers have been wildly used to allow the ligands to bend and rotate when it coordinates to metal centers. And the flexibility of ligands is essential to forming some particular properties and structures [8–10]. The secondary ligands are selected to tune up the structures of complexes [11]. It is well-known that multidentate O-donor organic aromatic carboxylate ligands, such isomeric aromatic dicarboxylate ligands as phthalate (1,2-benzenedicarboxylate, 1,2-bdc), isophthalate (1,3-bdc), or terephthalate (1,4-bdc) [12], have been extensively employed in the construction of a variety of high-dimensional structures [8]. The phenyl dicarboxylate isomers contain two carboxyl groups at distinct positions, which can engender significant spatial effect and thus influence the structural assembly with multiform coordination fashions.

The title complex crystallizes in the monoclinic (*C*2/*c*) space group, which consists of one half of a 1,4-bis(2-methylbenzimidazol)butane, one 4,4′-oxybis(benzoate) one water molecule, and one Cd(II) ion in the asymmetric unit. As shown in the figure, the center Cd(II) ion is five-coordinated, ligated by four oxygen atoms (O2, O3, O4 and O5) from four symmetry-related 4,4′-oxybis(benzoate), and one nitrogen atom (N1) from 1,4-bis(2-methylbenzimidazol)butane. The bond lengths are Zn(1)—O(2) = 2.211(2) Å, Zn(1)—O(3) = 2.237(2) Å, Zn(1)—O(4) = 2.241(3) Å, Zn(1)—O(5) = 2.218(3) Å and Zn(1)—N(1) = 2.237(2) Å, respectively. They are in the normal range [13].

Acknowledgements: This work was financially supported by the Science and technology research key project of the Education Department of Henan Province (2014A150025) and the Provincial cultivation fund of Luoyang normal university (2013-PYJJ-005).

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