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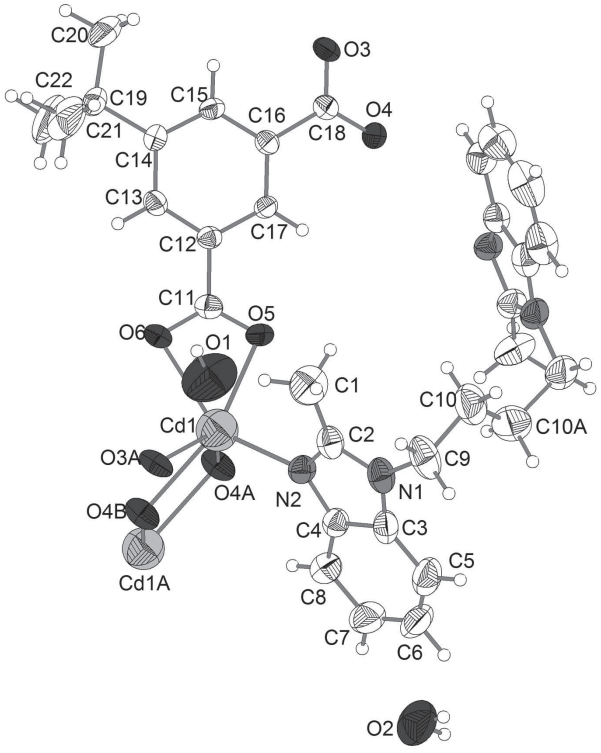
Crystal structure of poly[(μ_2-2-methyl-1-(4-(2-methyl-2*H*-benzo[*d*] imidazol-1(7a*H*)-yl)butyl)-1*H*-benzo[*d*]imidazole-κ^2*N*:*N*′)bis(μ_3-5-*tert*-butylbenzene-1,3-dicarboxylato-κ^4*O*:*O*,*O*′:*O*″, *O*‴)dicadmium(II)] tetrahydrate, $C_{44}H_{54}Cd_2N_4O_{12}$



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Abstract

$C_{44}H_{54}Cd_2N_4O_{12}$, monoclinic, $C2/c$, $a = 22.818(5)$ Å, $b = 10.714(2)$ Å, $c = 19.163(4)$ Å, $\beta = 102.43(3)°$, $V = 4575.2(16)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0381$, $wR_{ref}(F^2) = 0.1077$, $T = 293(2)$ K.

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The crystal structure is shown in the figure, Tables 1–3 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:	colourless, block, size 0.28×0.35×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.94 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15974, 4243
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3778
$N(param)_{refined}$:	284
Programs:	Bruker programs [4], SHELX [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1W)	8*f*	0.2523	0.6911	0.0344	0.171
H(2W)	8*f*	0.2051	0.6269	0.0268	0.171
H(3W)	8*f*	0.8128	0.6243	0.0356	0.140
H(4W)	8*f*	0.7666	0.5669	0.0337	0.140
H(1A)	8*f*	0.4003	0.6973	0.1814	0.101
H(1B)	8*f*	0.3665	0.6068	0.1219	0.101
H(1C)	8*f*	0.3743	0.7479	0.1042	0.101
H(5)	8*f*	0.5673	0.3701	0.0975	0.074
H(6)	8*f*	0.6452	0.4350	0.0463	0.081
H(7)	8*f*	0.6513	0.6362	0.0084	0.075
H(8)	8*f*	0.5794	0.7833	0.0211	0.058
H(9A)	8*f*	0.4727	0.3506	0.1374	0.077
H(9B)	8*f*	0.4113	0.4180	0.1343	0.077
H(10A)	8*f*	0.4503	0.5060	0.2499	0.084
H(10B)	8*f*	0.4498	0.3597	0.2532	0.084
H(13)	8*f*	0.3251	1.2195	0.1539	0.036
H(15)	8*f*	0.3193	1.2412	0.3598	0.034
H(17)	8*f*	0.4411	1.0200	0.2985	0.032
H(20A)	8*f*	0.2243	1.3113	0.3214	0.140

Table 2 (continued)

Atom	Site	x	y	z	U_{iso}
H(20B)	8*f*	0.2061	1.4402	0.2842	0.140
H(20C)	8*f*	0.2705	1.4215	0.3320	0.140
H(21A)	8*f*	0.1940	1.2102	0.2037	0.131
H(21B)	8*f*	0.2238	1.2499	0.1406	0.131
H(21C)	8*f*	0.1784	1.3397	0.1663	0.131
H(22A)	8*f*	0.2511	1.5118	0.1884	0.139
H(22B)	8*f*	0.2945	1.4261	0.1565	0.139
H(22C)	8*f*	0.3165	1.4871	0.2319	0.139

Source of material

A mixture of 5-*tert*-butylisophthalic acid (0.1 mmol, 0.020 g), $Cd(OAc)_2{\cdot}2H_2O$ (0.1 mmol, 0.238 g), THF (10 mL), NaOH solution (3 mL), 2-methyl-1-(4-(2-methyl-2*H*-benzo[*d*]imidazol-1(7a*H*)-yl)butyl)-1*H*-benzo[*d*]imidazole (0.1 mmol, 0.070 g), and H_2O (10 mL) was heated in a 25 mL Teflon-lined autoclave under autogenous pressure at 423 K for 4 days. After cooling to room temperature, colourless crystals were collected by filtration and washed with distilled water.

Experimental details

The hydrogen atoms were placed on calculated positions using the AFIX options 3, 23, 43 and 137 of the SHELX program [5].

Discussion

Benzimidazole and its derivatives are important building units of N-containing heterocyclic ring compounds, as they provide supramolecular recognition sites for N—H···O, C—H···O, C—H···π and π-π interactions. Multi-carboxylate ligands are extremely popular as efficient linkers in the construction of supramolecular compounds. The title compound contains a DI-Carboxylate!!!. Discrete subunits or low-dimensional entities have been linked into supramolecular compounds through hydrogen bonds, π···π stacking and host-guest interactions [1–3].

In this contribution, we have formed the interesting supramolecular network compound via hydrogen bonds and π···π stacking interactions. The title structure comprises a 2-methyl-1-(4-(2-methyl-2*H*-benzo[*d*]imidazol-

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	8*f*	0.45661(1)	0.92899(2)	0.06129(1)	0.0331(2)	0.0314(2)	0.0234(2)	−0.0004(1)	0.0091(1)	−0.0013(1)
N(1)	8*f*	0.4761(2)	0.5342(3)	0.1183(2)	0.057(2)	0.034(2)	0.043(2)	−0.007(2)	0.002(2)	0.006(2)
N(2)	8*f*	0.4818(2)	0.7285(3)	0.0812(2)	0.043(2)	0.030(2)	0.043(2)	−0.000(2)	0.012(2)	0.001(2)
O(1)	8*f*	0.2414(2)	0.6262(5)	0.0226(3)	0.077(3)	0.109(4)	0.171(5)	−0.016(3)	0.057(3)	0.001(4)
O(2)	8*f*	0.7737(2)	0.6295(5)	0.0158(2)	0.069(3)	0.105(4)	0.097(3)	0.024(3)	−0.003(2)	−0.003(3)
O(3)	8*f*	0.3803(1)	0.8700(3)	−0.0361(1)	0.042(2)	0.064(2)	0.024(1)	−0.013(1)	0.010(1)	−0.003(1)
O(4)	8*f*	0.5434(1)	1.0347(3)	0.0625(1)	0.038(2)	0.060(2)	0.030(2)	−0.021(1)	0.004(1)	−0.004(1)
O(5)	8*f*	0.4469(1)	0.9634(3)	0.1744(2)	0.050(2)	0.056(2)	0.030(2)	0.021(2)	0.011(1)	−0.004(1)
O(6)	8*f*	0.3962(2)	1.0911(3)	0.0953(2)	0.060(2)	0.064(2)	0.025(2)	0.020(2)	0.014(1)	0.003(1)
C(1)	8*f*	0.3928(2)	0.6777(5)	0.1314(3)	0.064(3)	0.056(3)	0.088(4)	−0.009(3)	0.030(3)	0.013(3)
C(2)	8*f*	0.4500(2)	0.6482(4)	0.1110(2)	0.047(3)	0.038(2)	0.042(2)	−0.004(2)	0.005(2)	0.006(2)
C(3)	8*f*	0.5275(2)	0.5406(4)	0.0911(2)	0.055(3)	0.036(2)	0.037(2)	0.005(2)	−0.004(2)	0.002(2)
C(4)	8*f*	0.5307(2)	0.6634(4)	0.0681(2)	0.038(2)	0.035(2)	0.037(2)	0.001(2)	0.004(2)	−0.001(2)
C(5)	8*f*	0.5698(3)	0.4523(5)	0.0829(3)	0.077(4)	0.040(3)	0.060(3)	0.015(2)	−0.003(3)	−0.003(2)
C(6)	8*f*	0.6158(3)	0.4919(6)	0.0520(3)	0.061(3)	0.070(4)	0.063(3)	0.035(3)	−0.002(3)	−0.014(3)
C(7)	8*f*	0.6196(2)	0.6133(6)	0.0291(3)	0.050(3)	0.076(4)	0.062(3)	0.013(3)	0.015(2)	−0.005(3)
C(8)	8*f*	0.5770(2)	0.7016(4)	0.0366(2)	0.046(3)	0.046(3)	0.052(3)	0.002(2)	0.010(2)	−0.004(2)
C(9)	8*f*	0.4544(3)	0.4251(4)	0.1519(3)	0.090(4)	0.045(3)	0.051(3)	−0.020(3)	0.002(3)	0.014(2)
C(10)	8*f*	0.4680(3)	0.4309(5)	0.2350(3)	0.091(4)	0.055(3)	0.062(4)	0.000(3)	0.014(3)	0.017(3)
C(11)	8*f*	0.4112(2)	1.0526(3)	0.1589(2)	0.027(2)	0.037(2)	0.029(2)	−0.004(2)	0.005(2)	−0.006(2)
C(12)	8*f*	0.3862(2)	1.1107(3)	0.2167(2)	0.029(2)	0.025(2)	0.030(2)	−0.002(1)	0.012(2)	0.000(2)
C(13)	8*f*	0.3394(2)	1.1954(3)	0.2011(2)	0.033(2)	0.031(2)	0.024(2)	−0.001(2)	0.004(2)	0.000(2)
C(14)	8*f*	0.3127(2)	1.2458(3)	0.2540(2)	0.029(2)	0.027(2)	0.033(2)	0.003(2)	0.005(2)	0.002(2)
C(15)	8*f*	0.3362(2)	1.2094(3)	0.3234(2)	0.032(2)	0.026(2)	0.029(2)	0.002(2)	0.010(2)	−0.005(2)
C(16)	8*f*	0.3845(2)	1.1264(3)	0.3412(2)	0.028(2)	0.029(2)	0.024(2)	−0.003(2)	0.004(1)	−0.002(2)
C(17)	8*f*	0.4094(2)	1.0761(3)	0.2875(2)	0.025(2)	0.027(2)	0.028(2)	0.004(1)	0.005(2)	−0.003(1)
C(18)	8*f*	0.4077(2)	1.0942(3)	0.4181(2)	0.031(2)	0.027(2)	0.028(2)	−0.004(2)	0.005(2)	−0.003(2)
C(19)	8*f*	0.2608(2)	1.3386(4)	0.2341(2)	0.038(2)	0.040(2)	0.040(2)	0.014(2)	0.003(2)	0.002(2)
C(20)	8*f*	0.2384(3)	1.3818(7)	0.2988(3)	0.092(5)	0.124(6)	0.063(4)	0.077(4)	0.013(3)	−0.001(4)
C(21)	8*f*	0.2096(3)	1.2792(6)	0.1814(3)	0.052(3)	0.103(5)	0.090(4)	0.031(3)	−0.023(3)	−0.023(4)
C(22)	8*f*	0.2827(3)	1.4512(5)	0.1996(4)	0.084(5)	0.052(3)	0.148(7)	0.028(3)	0.038(4)	0.040(4)

1(7a*H*)-yl)butyl)-1*H*-benzo[*d*]imidazole and a half of a 5-*tert*-butyl-isophthalate per asymmetric unit. The cadmium atom is six-coordinated in a distorted octahedral manner the Cd—O bond lengths range from 2.257(3)–2.403(3) Å. The bond angles of O—Cd—O are in the range of 54.36(9)° to 160.47(12)°. The coordination polymer is found to be chain-type. Several kinds of hydrogen bonding are observed in the crystal structure: the hydrogen bonding between oxygen atoms of carboxylate motif and uncoordinated water molecules (d(O···O): 2.844(5) Å and 2.910(5) Å, and O—H—O are 179.5° and 168.6°, respectively). In addition, there is a kind of intermolecular hydrogen bonds in the internal uncoordinated water molecules (d(O···O): 2.838(7) Å and 2.845(11) Å, and O—H—O are 124.1° and 134.7°, respectively). Weaker π-π interactions are observed between the 2-methyl-1-(4-(2-methyl-2*H*-benzo[*d*]imidazol-1(7a*H*)-yl)butyl)-1*H*-benzo[*d*]imidazole ligands with the centroid–centroid distance of 3.792 Å, further consolidating the stability of the structure.

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References

1. Weng, Z. H.; Liu, D. C.; Chen, Z. L.; Zou, H. H.; Qin, S. N.; Liang, F. P.: Two types of lanthanide coordination polymers of (2,3-*f*)-pyrazino(1,10)phenanthroline-2,3-dicarboxylic acid: syntheses, structures, and properties. Cryst. Growth Des. **9** (2009) 4163–4170.
2. Yang, J.; Ma, J. F.; Liu, Y. Y.; Ma, J. C.; Batten, S. R.: Organic-acid effect on the structures of a series of lead(II) complexes. Inorg. Chem. **46** (2007) 6542–6555.
3. Liu, C. B.; Chen, J.; Bai, H. Y.; Wang, S. T.; Wang, Q. W.; Che, G. B.: Hydrothermal synthesis, crystal structure and magnetic behavior of a novel 3D supramolecular complex $[Cu(HDPPZC)(SO_4)(H_2O)]_2$. Chin. J. Struct. Chem. **8** (2012) 1182–1186.
4. Bruker: SAINT-Plus, SHELXTL and SADABS. Bruker AXS Inc., Madison, WI, USA, 2004.
5. Sheldrick, G. M.: SHELXL97: Programs for structure solution and refinement. University of Göttingen, Germany (1997)