Crystal structure of *catena*-monoaqua(5-*tert*-butylisophthalate)-cadmium(II)-[1,4-*bis*-(2-methyl-imidazol-1-yl)butane]-hydrate (1:1:2), $[Cd(C_{12}H_{13}O_4)(C_{12}H_{12}O_4)(H_2O)]\cdot(C_{12}H_{20}N_4)_{0.5}\cdot 2H_2O$, $C_{30}H_{41}CdN_2O_{11}$

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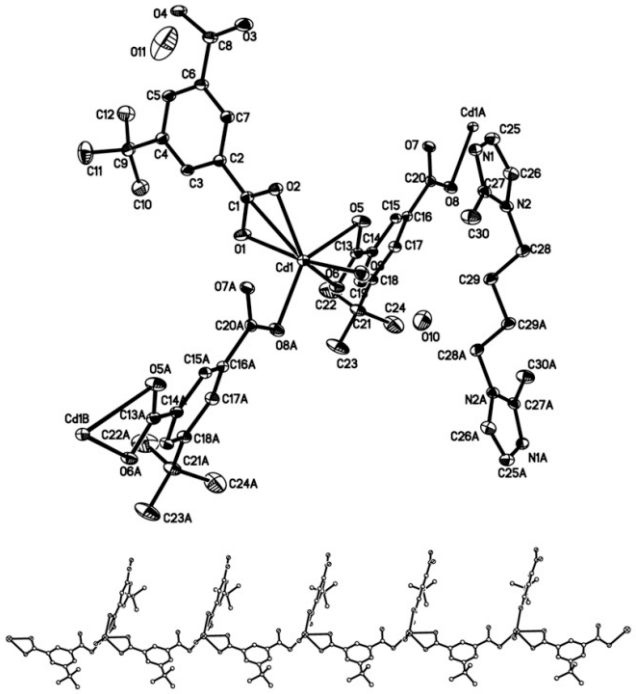

Abstract

$C_{30}H_{41}CdN_2O_{11}$, triclinic, $P\bar{1}$ (no. 2), a = 10.266(2) Å, b = 11.907(2) Å, c = 14.989(2) Å, α = 104.822(1)°, β = 108.566(1)°, γ = 95.923(2)°, V = 1644.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.0307, $wR_{ref}(F^2)$ = 0.0814, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.11×0.17×0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.23 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12302, 6077
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5514
$N(param)_{refined}$:	418
Programs:	SHELXS-97, SHELXL-97 [9]

Source of material

The title compound was prepared under the hydrothermal conditions. A mixture of 5-*tert*-butylisophthalate (0.2 mmol) and 1,4-*bis*-(2-methyl-imidazol-1-yl)butane (0.1 mmol), $Cd(NO_3)_2$ (0.1 mmol), NaOH (0.1mmol) and H_2O (15 mL) were placed in a Teflon-lined stainless steel vessel, heated to 440 K for 4 day, and then cooled to room temperature within 24 h. Colourless block crystals of the title complex were obtained.

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Discussion

Designing solids with molecules that encode well defined non-covalent interactions has recently become a rapidly growing area of research due to their fascinating molecular and/or supramolecular structural diversity and potential applications for catalysis [1-4] and material sciences [5-6]. It is well known that carboxylate ligands play an important role in coordination chemistry, which may adopt diverse binding modes such as monodentate, chelating, and bridging in the *syn-syn*, *syn-anti*, and *anti-anti* configurations [7-8]. 5-*tert*-butylisophthalate ($C(CH_3)_3$-ip) acid, possesses several interesting characteristics: (a) it has two carboxyl groups which may be completely or partially deprotonated, inducing rich coordination modes and allowing interesting structures with higher dimensions, (b) it can act not only as hydrogen-bond acceptor but also as hydrogen-bond donor, depending upon the numbers of deprotonated carboxyl groups. In this paper, we select 5-*tert*-butylisophthalate ($C(CH_3)_3$-ip) and 1,4-*bis*-(2-methyl-imidazol-1-yl)butane (bib) to react with Cd(II) ions to obtain a new MOFs. The X-ray structural analysis shows that the title complex crystallizes in the triclinic system, space group $P\bar{1}$. The asymmetric structure unit of the complex consists of a Cd^{2+} ion, two $C(CH_3)_3$-ip ligands, a half free bib ligand, a coordinated water molecule and two free water molecule (figure, top). The Cd^{2+} ion is chelated by six oxygen atoms from three carboxylate groups of three $C(CH_3)_3$-ip ligands forming three chelating rings, and one oxygen atom from a water molecule complete the sevenfold-coordination at the metal center. Thus, the coordination geometry around Cd^{2+} ion can be described as a strongly distorted pentagonal bipyramid. Five carboxylate oxygen atoms (O2, O5, O6, O7A, O8A) comprise the equatorial plane around Cd^{2+} ion with a maximum deviation of 1.33 Å (for O2) from the rough plane, while the other two oxygen atoms (O1, O9) form the pyramidal apices with bond angles O1-Cd1-O9 of 169.33(8)°. The Cd–O bond lengths are in the range of 2.245(2) - 2.287(2) Å. It is worthwhile to note that the $C(CH_3)_3$-ip ligands adopt two kinds of coordination modes in the crystal structure. One CH_3O-ip ligand has a bidentate-chelating coordination mode to bridge the neighbouring Cd^{2+} ions to yield a 1D linear chain (figure, bottom), with a Cd···Cd distance of 10.266(2) Å. The other CH_3O-ip ligand is partially deprotonated and adopts a monodentate coordination mode, which serves to complete the geometry coordination sphere of the Cd^{2+} ion. The bib ligands are protonated and act as hydrogen-bond donors to CH_3O-ip ligands (O7 atoms). As a consequence, the 1D chains are connected to a 2D layers, which are further assembled by hy-

drogen bonds between coordinated or free water molecules and $C(CH_3)_3$-ip ligands to build a three-dimensional supramolecular structure. The bond lengths and angles of these hydrogen bonding parametres are in the range of 2.567(3) - 3.426(7) Å and 165.5 - 179.6°, respectively.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(10A)	2*i*	0.703	0.8017	0.6002	1.1122	0.106
H(10B)	2*i*	0.703	0.9385	0.5788	1.0908	0.106
H(10C)	2*i*	0.703	0.9253	0.5713	1.1906	0.106
H(11A)	2*i*	0.703	0.6602	0.3027	1.0767	0.137
H(11B)	2*i*	0.703	0.6186	0.4267	1.0825	0.137
H(11C)	2*i*	0.703	0.7261	0.4074	1.1758	0.137
H(12A)	2*i*	0.703	0.9643	0.3828	1.1853	0.1
H(12B)	2*i*	0.703	1.0091	0.379	1.0941	0.1
H(12C)	2*i*	0.703	0.8976	0.2746	1.0887	0.1
H(10D)	2*i*	0.297	0.5997	0.3967	1.0536	0.106
H(10E)	2*i*	0.297	0.6673	0.5317	1.0865	0.106
H(10F)	2*i*	0.297	0.6946	0.4621	1.1641	0.106
H(11D)	2*i*	0.297	0.8971	0.3657	1.1915	0.137
H(11E)	2*i*	0.297	0.9310	0.3014	1.0988	0.137
H(11F)	2*i*	0.297	0.7816	0.2676	1.1025	0.137
H(12D)	2*i*	0.297	0.8857	0.5976	1.0895	0.1
H(12E)	2*i*	0.297	1.0031	0.5222	1.0979	0.1
H(12F)	2*i*	0.297	0.9514	0.564	1.1865	0.1
H(4)	2*i*		0.7428	–0.0185	0.7597	0.076
H(1W)	2*i*		0.4545	0.638	0.4464	0.069
H(2W)	2*i*		0.5122	0.7465	0.4946	0.069
H(3W)	2*i*		0.6185	0.9188	0.4494	0.137
H(4W)	2*i*		0.6434	0.9507	0.5434	0.137
H(5W)	2*i*		0.4023	–0.0266	0.6775	0.282
H(6W)	2*i*		0.4777	0.0409	0.6982	0.282
H(1)	2*i*		0.8679	0.4821	0.3792	0.059
H(3)	2*i*		0.7139	0.5438	0.9437	0.046
H(5)	2*i*		0.7869	0.2185	0.9369	0.049
H(7)	2*i*		0.6462	0.3008	0.6838	0.046
H(15)	2*i*		1.0359	0.7003	0.6298	0.035
H(17)	2*i*		1.3594	0.9517	0.8328	0.042
H(19)	2*i*		0.9687	0.9678	0.8241	0.04
H(22A)	2*i*		1.3832	1.013	1.0057	0.169
H(22B)	2*i*		1.2564	1.0122	1.0425	0.169
H(22C)	2*i*		1.3684	1.1293	1.0763	0.169
H(23A)	2*i*		1.0634	1.1023	0.9777	0.132
H(23B)	2*i*		1.0615	1.1679	0.8991	0.132
H(23C)	2*i*		1.1623	1.2262	1.0093	0.132
H(24A)	2*i*		1.3646	1.2519	0.9704	0.143
H(24B)	2*i*		1.2791	1.1997	0.8567	0.143
H(24C)	2*i*		1.4077	1.1473	0.9057	0.143
H(25)	2*i*		1.1178	0.5124	0.4124	0.054
H(26)	2*i*		1.1915	0.7202	0.427	0.055
H(28A)	2*i*		0.8881	0.8582	0.3539	0.055
H(28B)	2*i*		1.0501	0.8836	0.3763	0.055
H(29A)	2*i*		1.0925	0.9336	0.5462	0.053
H(29B)	2*i*		0.9303	0.9085	0.5237	0.053
H(30A)	2*i*		0.7205	0.7205	0.4187	0.095
H(30B)	2*i*		0.6678	0.5875	0.3528	0.095
H(30C)	2*i*		0.6922	0.6869	0.3051	0.095

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(10)	2*i*	0.703	0.8752(9)	0.5557(4)	1.1212(5)	0.104(4)	0.058(3)	0.037(3)	0.012(3)	0.014(3)	0.008(2)
C(11)	2*i*	0.703	0.6935(7)	0.3869(9)	1.1050(5)	0.105(4)	0.117(5)	0.053(3)	0.005(4)	0.033(3)	0.029(3)
C(12)	2*i*	0.703	0.9325(8)	0.3588(6)	1.1144(4)	0.087(4)	0.072(3)	0.035(3)	0.031(3)	0.008(3)	0.017(2)
C(10')	2*i*	0.297	0.681(1)	0.457(1)	1.097(1)	0.104(4)	0.058(3)	0.037(3)	0.012(3)	0.014(3)	0.008(2)
C(11')	2*i*	0.297	0.860(2)	0.331(1)	1.121(1)	0.105(4)	0.117(5)	0.053(3)	0.005(4)	0.033(3)	0.029(3)
C(12')	2*i*	0.297	0.923(1)	0.537(1)	1.116(1)	0.087(4)	0.072(3)	0.035(3)	0.031(3)	0.008(3)	0.017(2)
Cd(1)	2*i*		0.54929(2)	0.68044(2)	0.65283(2)	0.0285(1)	0.0261(1)	0.0314(1)	0.00760(8)	0.00974(9)	0.00832(8)
O(1)	2*i*		0.6355(3)	0.6300(2)	0.8044(2)	0.086(2)	0.031(1)	0.037(1)	0.021(1)	0.010(1)	0.007(1)
O(2)	2*i*		0.5726(2)	0.4938(2)	0.6607(2)	0.048(1)	0.031(1)	0.030(1)	0.0098(9)	0.0069(9)	0.0089(9)
O(3)	2*i*		0.7156(4)	0.0965(2)	0.6589(2)	0.156(3)	0.043(2)	0.044(2)	0.035(2)	0.039(2)	0.009(1)
O(4)	2*i*		0.7366(3)	0.0450(2)	0.7941(2)	0.078(2)	0.032(1)	0.043(1)	0.024(1)	0.020(1)	0.010(1)
O(5)	2*i*		0.7829(2)	0.6703(2)	0.6182(2)	0.032(1)	0.032(1)	0.068(2)	0.0060(9)	0.011(1)	–0.002(1)
O(6)	2*i*		0.7401(2)	0.8307(2)	0.7057(2)	0.027(1)	0.031(1)	0.055(1)	0.0098(8)	0.016(1)	0.0075(9)
O(7)	2*i*		1.2855(2)	0.6440(2)	0.6485(2)	0.041(1)	0.034(1)	0.047(1)	0.0129(9)	0.022(1)	0.009(1)
O(8)	2*i*		1.4322(2)	0.8162(2)	0.7057(2)	0.031(1)	0.040(1)	0.059(1)	0.0069(9)	0.023(1)	0.006(1)
O(9)	2*i*		0.4563(2)	0.6908(2)	0.4975(2)	0.052(1)	0.052(1)	0.031(1)	0.016(1)	0.012(1)	0.008(1)
O(10)	2*i*		0.6165(4)	0.8880(3)	0.4943(2)	0.149(3)	0.052(2)	0.058(2)	–0.002(2)	0.034(2)	0.004(1)
O(11)	2*i*		0.3921(7)	0.0390(6)	0.6664(5)	0.177(5)	0.202(5)	0.219(6)	0.025(4)	0.063(4)	0.136(5)
N(1)	2*i*		0.9190(3)	0.5482(2)	0.3867(2)	0.047(2)	0.027(1)	0.042(2)	0.005(1)	0.018(1)	0.007(1)
N(2)	2*i*		0.9825(3)	0.7306(2)	0.3979(2)	0.043(2)	0.027(1)	0.040(1)	0.005(1)	0.017(1)	0.005(1)
C(1)	2*i*		0.6241(3)	0.5261(3)	0.7546(2)	0.035(2)	0.031(2)	0.034(2)	0.009(1)	0.009(1)	0.010(1)
C(2)	2*i*		0.6712(3)	0.4360(3)	0.8056(2)	0.039(2)	0.031(2)	0.032(2)	0.010(1)	0.011(1)	0.009(1)
C(3)	2*i*		0.7156(3)	0.4676(3)	0.9085(2)	0.056(2)	0.027(1)	0.029(2)	0.011(1)	0.014(1)	0.005(1)
C(4)	2*i*		0.7626(4)	0.3881(3)	0.9605(2)	0.058(2)	0.033(2)	0.030(2)	0.011(1)	0.014(1)	0.009(1)
C(5)	2*i*		0.7602(4)	0.2743(3)	0.9048(2)	0.059(2)	0.032(2)	0.033(2)	0.015(1)	0.013(2)	0.012(1)
C(6)	2*i*		0.7187(3)	0.2416(3)	0.8020(2)	0.049(2)	0.029(2)	0.033(2)	0.013(1)	0.011(1)	0.007(1)
C(7)	2*i*		0.6740(3)	0.3224(3)	0.7523(2)	0.050(2)	0.033(2)	0.027(2)	0.011(1)	0.009(1)	0.006(1)
C(8)	2*i*		0.7219(4)	0.1210(3)	0.7437(2)	0.055(2)	0.034(2)	0.035(2)	0.016(2)	0.012(2)	0.007(1)
C(9)	2*i*		0.8118(4)	0.4242(3)	1.0738(2)	0.084(3)	0.041(2)	0.028(2)	0.012(2)	0.018(2)	0.011(1)
C(13)	2*i*		0.8245(3)	0.7721(2)	0.6766(2)	0.029(1)	0.028(1)	0.040(2)	0.008(1)	0.011(1)	0.013(1)
C(14)	2*i*		0.9778(3)	0.8271(2)	0.7195(2)	0.028(1)	0.027(1)	0.034(2)	0.009(1)	0.010(1)	0.011(1)
C(15)	2*i*		1.0701(3)	0.7697(2)	0.6829(2)	0.032(1)	0.024(1)	0.031(1)	0.007(1)	0.011(1)	0.007(1)
C(16)	2*i*		1.2132(3)	0.8162(2)	0.7255(2)	0.032(2)	0.028(1)	0.034(2)	0.010(1)	0.014(1)	0.011(1)
C(17)	2*i*		1.2635(3)	0.9206(3)	0.8050(2)	0.026(1)	0.033(2)	0.042(2)	0.004(1)	0.010(1)	0.007(1)
C(18)	2*i*		1.1739(3)	0.9795(3)	0.8440(2)	0.034(2)	0.030(2)	0.038(2)	0.007(1)	0.011(1)	0.005(1)
C(19)	2*i*		1.0308(3)	0.9304(2)	0.7995(2)	0.031(2)	0.031(2)	0.039(2)	0.011(1)	0.015(1)	0.006(1)
C(20)	2*i*		1.3159(3)	0.7541(3)	0.6902(2)	0.031(2)	0.036(2)	0.031(2)	0.012(1)	0.013(1)	0.012(1)
C(21)	2*i*		1.2331(3)	1.0916(3)	0.9345(3)	0.040(2)	0.037(2)	0.048(2)	0.007(1)	0.010(2)	–0.006(2)

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(22)	2*i*		1.3180(7)	1.0585(5)	1.0227(4)	0.158(5)	0.090(4)	0.051(3)	0.048(4)	0.001(3)	–0.010(2)
C(23)	2*i*		1.1197(5)	1.1526(4)	0.9573(4)	0.064(3)	0.061(3)	0.102(4)	0.007(2)	0.029(3)	–0.034(2)
C(24)	2*i*		1.3301(6)	1.1809(4)	0.9150(4)	0.093(4)	0.051(2)	0.118(4)	–0.018(2)	0.050(3)	–0.018(3)
C(25)	2*i*		1.0605(4)	0.5677(3)	0.4059(2)	0.045(2)	0.040(2)	0.047(2)	0.016(2)	0.014(2)	0.011(2)
C(26)	2*i*		1.1008(4)	0.6815(3)	0.4136(3)	0.038(2)	0.040(2)	0.054(2)	0.005(1)	0.016(2)	0.005(2)
C(27)	2*i*		0.8730(3)	0.6479(3)	0.3822(2)	0.043(2)	0.031(2)	0.037(2)	0.006(1)	0.016(1)	0.003(1)
C(28)	2*i*		0.9785(4)	0.8553(3)	0.3992(3)	0.064(2)	0.027(2)	0.048(2)	0.006(2)	0.023(2)	0.011(1)
C(29)	2*i*		1.0021(4)	0.9360(3)	0.5006(2)	0.056(2)	0.028(2)	0.046(2)	0.007(1)	0.016(2)	0.011(1)
C(30)	2*i*		0.7253(4)	0.6619(4)	0.3630(3)	0.043(2)	0.052(2)	0.091(3)	0.011(2)	0.027(2)	0.011(2)

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