Crystal structure of aqua[1,4-bis((1*H*-imidazol-1-yl)methyl)benzene]-(5-hydroxyisophthalato)cadmium(II), $Cd(H_2O)(C_{14}H_{14}N_4)(C_8H_4O_5)$

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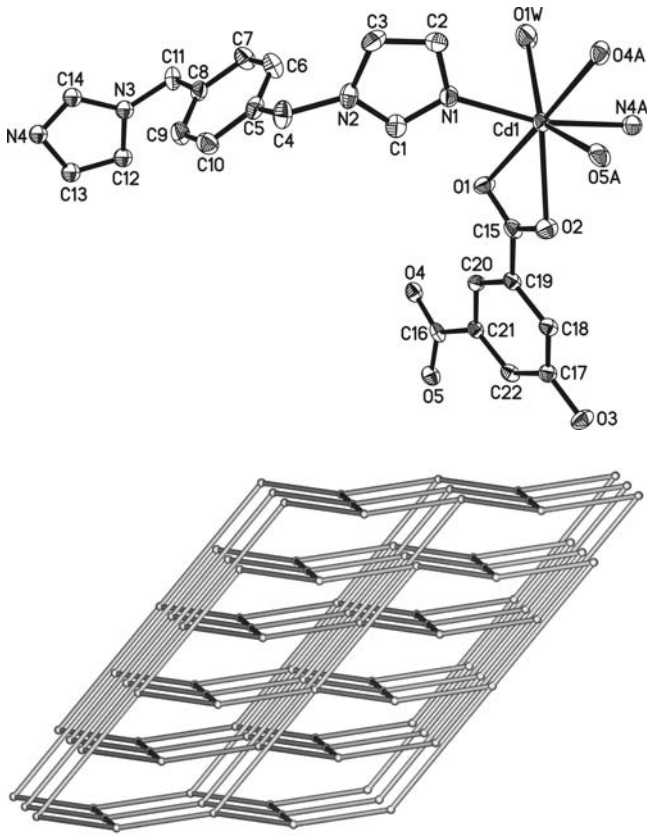

Abstract

$C_{22}H_{20}CdN_4O_6$, *Pn* (no. 7), a = 11.5679(5) Å, b = 8.4250(3) Å, c = 11.6371(4) Å, β = 113.390(5)°, V = 1041.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.022, $wR_{ref}(F^2)$ = 0.045, T = 293 K.

Source of material

All reagents and solvents were purchased from commercial sources and used without purification except 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene which was prepared according to [1]. A mixture of 5-hydroxyisophthalic acid (0.018 g, 0.1 mmol), $Cd(OH)_2$ (0.015 g, 0.1 mmol), 1,4-bis((1*H*-imidazol-1-yl)-methyl)benzene (0.024 g, 0.1 mmol) and 8 mL water was sealed in a 20 mL Teflon-lined reactor, which was heated at 130 °C for 3 days and then cooled to ambient temperature at 10 °C/h. Colorless block-shaped crystals of the title compound were collected in 38 % yield.

Experimental details

All H atoms on C atoms and on hydroxy O atoms were generated geometrically and refined as riding with d(C—H) = 0.93 - 0.97 Å, d(O—H) = 0.82 Å, and U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(O). H atoms of water molecule were located from difference Fourier maps and refined as riding with d(O—H) = 0.840 - 0.847 Å, U_{iso}(H) = 1.5 U_{eq}(O). The Flack parameter is –0.001(19), this provides evidence that the absolute structure has been assigned correctly.

Discussion

In the title crystal structure, there are one Cd(II) ion, one 5-hydroxyisophthalate anion, one 1,4-bis((1*H*-imidazol-1-yl)-methyl)benzene molecule and one coordinating water molecule in the asymmetric unit (figure, top). Each Cd(II) exhibits a pentagonal bipyramid coordination, defined by four carboxylate oxygen atoms from two different 5-hydroxyisophthalate anions (d(Cd1—O1) = 2.552(3) Å, d(Cd1—O2) = 2.374(3) Å, d(Cd1—O4A) = 2.412(3) Å and d(Cd1—O5A) = 2.500(3) Å), two nitrogen atoms from two individual 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene molecules (d(Cd1—N1) = 2.237(3) Å and d(Cd1—N4A) = 2.286(3) Å) and one oxygen atom from coordinating water molecule (d(Cd1—O1W) = 2.459(3) Å). Each 5-hydroxyisophthalate anion links two Cd(II) ions to form a wave-like chain with d(Cd···Cd) = 9.8384(7) Å through the carboxylate group in a mono-dentate chelate mode. The chains are further linked by the 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene molecules to form a complicated 3D framework. From a topological point of view, if the Cd(II) center is considered as the 4-connecting node, the structure of the title compound can be described as a uninodal 4-connected 3D (6^5.8) $CdSO_4$-type framework. Two identical networks form a 2-fold interpenetrating framework (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.22 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.99 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.5°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4613, 3226
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2883
$N(param)_{refined}$:	304
Programs:	SHELXS-97, SHELXL-97 [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3')	2*a*	0.8021	0.6802	0.8087	0.066
H(22)	2*a*	0.8007	0.7283	0.5256	0.037
H(20)	2*a*	0.4507	0.5552	0.3443	0.035
H(18)	2*a*	0.6036	0.5756	0.7195	0.036
H(3)	2*a*	–0.0369	–0.1413	0.3910	0.051
H(6)	2*a*	–0.0046	0.0082	0.1485	0.057
H(1)	2*a*	0.2568	0.1141	0.4137	0.045
H(2)	2*a*	–0.0368	0.1065	0.5002	0.049
H(11A)	2*a*	–0.0426	–0.0803	–0.2548	0.049
H(11B)	2*a*	0.0560	0.0576	–0.2242	0.049
H(4A)	2*a*	0.1190	–0.2794	0.3136	0.054
H(4B)	2*a*	0.2537	–0.2040	0.3592	0.054
H(9)	2*a*	0.2349	–0.2294	–0.0383	0.054
H(7)	2*a*	–0.0496	0.0536	–0.0594	0.053
H(10)	2*a*	0.2822	–0.2693	0.1714	0.053
H(13)	2*a*	0.3331	–0.2287	–0.3496	0.047
H(14)	2*a*	0.0238	–0.3528	–0.3138	0.043
H(12)	2*a*	0.2657	–0.0026	–0.2586	0.046
H(1A)	2*a*	–0.036(4)	0.484(5)	0.325(1)	0.057
H(1B)	2*a*	–0.074(3)	0.535(5)	0.423(3)	0.057

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*a*	0.17403(3)	0.40135(3)	0.57465(3)	0.0320(1)	0.0345(1)	0.0254(1)	–0.0031(2)	0.01627(9)	–0.0039(2)
O(3)	2*a*	0.8178(2)	0.7013(4)	0.7476(2)	0.035(2)	0.070(2)	0.024(2)	–0.011(2)	0.008(1)	–0.001(2)
O(2)	2*a*	0.3936(2)	0.4491(3)	0.6442(2)	0.037(2)	0.065(2)	0.031(2)	–0.009(1)	0.021(1)	–0.001(1)
O(1)	2*a*	0.2992(3)	0.4537(4)	0.4408(3)	0.028(2)	0.061(2)	0.026(2)	–0.007(2)	0.009(1)	–0.004(2)
C(17)	2*a*	0.7184(4)	0.6611(5)	0.6419(4)	0.030(2)	0.039(2)	0.027(2)	0.002(2)	0.012(2)	0.002(2)
N(2)	2*a*	0.1262(4)	–0.0580(4)	0.3814(3)	0.051(2)	0.032(2)	0.025(2)	–0.001(2)	0.015(2)	–0.001(2)
C(8)	2*a*	0.0854(4)	–0.0852(4)	–0.0733(4)	0.039(2)	0.026(2)	0.039(2)	–0.004(2)	0.016(2)	–0.006(2)
C(22)	2*a*	0.7277(4)	0.6846(5)	0.5275(4)	0.031(2)	0.035(2)	0.034(2)	0.003(2)	0.019(2)	0.008(2)
C(20)	2*a*	0.5183(3)	0.5795(4)	0.4187(3)	0.028(2)	0.039(2)	0.022(2)	0.002(2)	0.009(2)	0.001(2)
C(18)	2*a*	0.6097(4)	0.5936(5)	0.6433(4)	0.030(2)	0.040(2)	0.025(2)	0.005(2)	0.015(2)	0.006(2)
C(5)	2*a*	0.1409(5)	–0.1386(6)	0.1815(5)	0.049(3)	0.030(2)	0.039(3)	–0.009(2)	0.019(3)	–0.008(2)
N(1)	2*a*	0.1247(3)	0.1668(4)	0.4775(3)	0.037(2)	0.035(2)	0.031(2)	–0.004(2)	0.017(2)	–0.006(1)
C(21)	2*a*	0.6293(3)	0.6435(4)	0.4165(3)	0.032(2)	0.033(2)	0.023(2)	0.007(2)	0.015(2)	0.003(2)
C(3)	2*a*	0.0226(4)	–0.0605(5)	0.4094(4)	0.037(2)	0.047(3)	0.039(2)	–0.013(2)	0.010(2)	–0.005(2)
C(19)	2*a*	0.5103(3)	0.5526(4)	0.5337(3)	0.030(2)	0.036(2)	0.024(2)	0.007(2)	0.013(2)	0.005(2)
C(6)	2*a*	0.0438(4)	–0.0409(5)	0.1112(4)	0.056(3)	0.044(2)	0.050(3)	0.012(2)	0.028(2)	–0.001(2)
C(1)	2*a*	0.1840(4)	0.0815(4)	0.4227(4)	0.045(2)	0.034(2)	0.038(2)	–0.004(2)	0.021(2)	0.000(2)
C(2)	2*a*	0.0229(4)	0.0772(5)	0.4693(4)	0.030(2)	0.051(3)	0.042(2)	0.000(2)	0.015(2)	–0.003(2)
C(11)	2*a*	0.0461(4)	–0.0546(4)	–0.2117(4)	0.058(3)	0.031(2)	0.040(2)	–0.000(2)	0.025(2)	–0.007(2)
C(4)	2*a*	0.1646(5)	–0.1825(5)	0.3141(4)	0.067(3)	0.027(2)	0.042(3)	0.003(2)	0.022(2)	–0.004(2)
C(9)	2*a*	0.1858(5)	–0.1812(7)	–0.0014(5)	0.049(3)	0.046(3)	0.046(3)	0.003(3)	0.026(3)	–0.010(3)
C(7)	2*a*	0.0165(4)	–0.0139(5)	–0.0141(4)	0.047(3)	0.046(2)	0.040(3)	0.013(2)	0.019(2)	0.004(2)
C(15)	2*a*	0.3940(4)	0.4790(5)	0.5391(4)	0.034(2)	0.041(2)	0.030(2)	0.005(2)	0.020(2)	0.002(2)
O(4)	2*a*	0.5480(3)	0.6466(4)	0.1940(3)	0.045(2)	0.040(2)	0.028(2)	0.007(2)	0.018(2)	0.004(2)
O(5)	2*a*	0.7524(3)	0.6878(3)	0.2976(2)	0.041(2)	0.050(2)	0.040(2)	–0.001(1)	0.028(2)	0.001(1)
C(16)	2*a*	0.6436(4)	0.6620(4)	0.2945(4)	0.042(2)	0.024(2)	0.034(2)	0.008(2)	0.025(2)	0.006(2)
C(10)	2*a*	0.2137(4)	–0.2062(6)	0.1246(4)	0.040(3)	0.044(3)	0.045(3)	0.007(2)	0.015(2)	0.002(2)
O(1W)	2*a*	–0.0201(3)	0.4910(4)	0.4028(3)	0.051(2)	0.084(2)	0.031(2)	0.020(2)	0.008(2)	–0.008(2)
N(3)	2*a*	0.1173(3)	–0.1451(3)	–0.2678(3)	0.044(2)	0.030(2)	0.032(2)	–0.003(2)	0.016(2)	0.001(2)
N(4)	2*a*	0.1750(3)	–0.3453(4)	–0.3548(3)	0.034(2)	0.032(2)	0.035(2)	–0.004(1)	0.013(2)	–0.001(1)
C(13)	2*a*	0.2613(4)	–0.2251(5)	–0.3323(4)	0.033(2)	0.045(2)	0.039(2)	–0.008(2)	0.012(2)	0.000(2)
C(14)	2*a*	0.0921(4)	–0.2935(4)	–0.3127(3)	0.042(2)	0.029(2)	0.041(2)	–0.008(2)	0.021(2)	–0.001(2)
C(12)	2*a*	0.2251(5)	–0.1000(5)	–0.2808(4)	0.044(3)	0.030(2)	0.037(3)	–0.011(2)	0.012(2)	0.001(2)

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