Crystal structure of (1,4-bis((1*H*-imidazol-1-yl)methyl)benzene)-(5-hydroxyisophthalato)zinc(II), $Zn(C_{14}H_{14}N_4)(C_8H_4O_5)$

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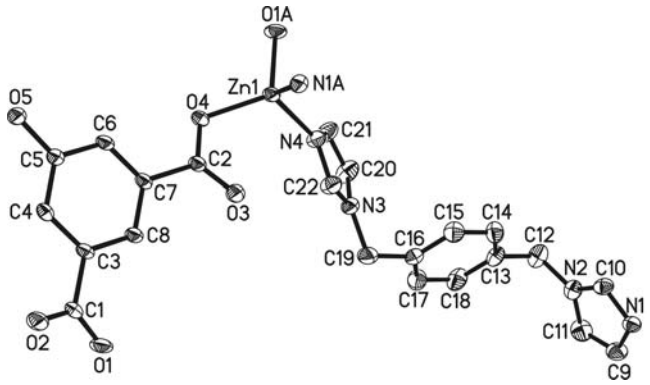

Abstract

$C_{22}H_{18}N_4O_5Zn$, monoclinic, $P2_1/n$ (no. 14), a = 8.1529(4) Å, b = 9.6506(4) Å, c = 25.706(1) Å, β = 97.864(4)°, V = 2003.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.064, T = 293 K.

Source of material

All reagents and solvents were purchased from commercial sources and used as received 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), $Zn(OH)_2$ (0.01 g, 0.1 mmol) and 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene (0.024 g, 0.1 mmol) was added to 4 mL ethanol and 4 mL water with stirring at room temperature. After a few minutes, the solution was transfered to a 15 mL Teflon-lined reactor, which was heated at 120 °C for 3 days and then cooled to room temperature at 10 °C/h. Colorless block-shaped crystals of the title compound were collected in 25 % yield.

Experimental details

All H atoms on C and 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), U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

The asymmetric unit contains one Zn(II) ion, one 5-hydroxyisophthalate anion and one 1,4-bis((1*H*-imidazol-1-yl)methyl)-benzene molecule. Zn(II) is four-coordinated by two N atoms from two different 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene units with d(Zn—N1) = 2.004 Å, d(Zn—N4) = 1.993 Å and two O atoms from two 5-hydroxyisophthalate anions with d(Zn—O1) = 1.971 Å, d(Zn—O4) = 1.968 Å in a tetrahedral manner. The 1,4-bis((1*H*-imidazol-1-yl)methyl)benzene molecules serve as bridges linking adjacent Zn centers into chains. The chains are further linked by the 5-hydroxyisophthalate anions via Zn—O bond to form a layer. The hydroxyl group of 5-hydroxyisophthalate is not deprotonated and does not participate in the coordinative interaction.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.20 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.71 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.3°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8857, 4626
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2583
$N(param)_{refined}$:	289
Programs:	SHELXS-97, SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(5A)	4*e*	0.4221	–0.2458	0.4655	0.080
H(5)	4*e*	0.4638	–0.0186	0.5624	0.041
H(12A)	4*e*	1.1042	0.0183	0.5758	0.056
H(12B)	4*e*	1.2500	0.1233	0.5895	0.056
H(7)	4*e*	0.5634	–0.3008	0.6772	0.038
H(3)	4*e*	0.4812	–0.4202	0.5263	0.050
H(15)	4*e*	0.8438	0.4276	0.4704	0.057
H(17)	4*e*	1.1611	0.1828	0.4096	0.065
H(10)	4*e*	1.0335	0.3115	0.7308	0.057
H(14)	4*e*	0.9163	0.3263	0.5513	0.058
H(19A)	4*e*	0.8356	0.4230	0.3775	0.076
H(19B)	4*e*	1.0188	0.4741	0.3797	0.076
H(18)	4*e*	1.2275	0.0764	0.4901	0.060
H(22)	4*e*	0.9211	0.0603	0.2574	0.056
H(20)	4*e*	1.1203	0.4230	0.2956	0.049
H(9)	4*e*	0.8140	0.1163	0.6075	0.048
H(21)	4*e*	0.8265	0.1341	0.3398	0.066
H(11)	4*e*	1.2441	0.2573	0.6748	0.059

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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}
Zn(01)	4*e*	0.63247(3)	0.24674(2)	0.690186(9)	0.0468(2)	0.0231(1)	0.0280(1)	0.0022(1)	0.0107(1)	–0.0006(1)
O(5)	4*e*	0.4256(2)	–0.1767(2)	0.48417(6)	0.099(2)	0.0320(9)	0.0257(9)	0.0059(9)	–0.005(1)	–0.0021(7)
O(4)	4*e*	0.5213(2)	0.0970(1)	0.64693(6)	0.056(1)	0.0236(8)	0.0323(9)	0.0019(7)	0.0030(8)	–0.0056(7)
O(3)	4*e*	0.6064(2)	–0.0514(2)	0.71118(6)	0.103(2)	0.0325(9)	0.0266(9)	0.0034(9)	0.006(1)	–0.0029(7)
O(1)	4*e*	0.5664(2)	–0.5642(1)	0.66708(6)	0.082(1)	0.0273(8)	0.0308(9)	0.0095(8)	0.0111(9)	0.0048(7)
O(2)	4*e*	0.5844(3)	–0.6164(2)	0.58500(7)	0.212(3)	0.031(1)	0.037(1)	0.027(1)	0.004(1)	–0.0046(9)
N(2)	4*e*	1.0537(2)	0.1670(2)	0.62513(7)	0.043(1)	0.050(1)	0.025(1)	0.0069(9)	0.010(1)	–0.0003(9)
N(1)	4*e*	0.8652(2)	0.2177(2)	0.67554(7)	0.044(1)	0.038(1)	0.030(1)	0.0023(9)	0.0097(9)	0.0000(8)
C(8)	4*e*	0.5502(3)	–0.0261(2)	0.66522(9)	0.047(2)	0.024(1)	0.033(1)	0.001(1)	0.014(1)	–0.002(1)
N(3)	4*e*	0.9685(3)	0.3080(2)	0.33374(7)	0.066(2)	0.047(1)	0.028(1)	0.005(1)	0.015(1)	0.0065(9)
C(5)	4*e*	0.4794(3)	–0.1104(2)	0.57298(8)	0.049(2)	0.022(1)	0.031(1)	0.004(1)	0.007(1)	0.001(1)
C(6)	4*e*	0.5191(3)	–0.1413(2)	0.62569(8)	0.042(1)	0.021(1)	0.027(1)	0.002(1)	0.008(1)	–0.0018(9)
N(4)	4*e*	1.0513(2)	0.2444(2)	0.25978(6)	0.050(1)	0.0323(9)	0.0252(9)	–0.003(1)	0.0071(8)	0.0056(9)
C(2)	4*e*	0.5275(3)	–0.3839(2)	0.60440(9)	0.053(2)	0.024(1)	0.027(1)	0.003(1)	0.004(1)	–0.001(1)
C(12)	4*e*	1.1307(3)	0.1159(3)	0.58068(9)	0.051(2)	0.060(2)	0.032(1)	0.013(1)	0.011(1)	–0.002(1)
C(7)	4*e*	0.5406(3)	–0.2793(2)	0.64172(8)	0.043(1)	0.028(1)	0.025(1)	0.0016(9)	0.008(1)	0.0015(9)
C(13)	4*e*	1.0796(3)	0.1899(2)	0.52937(9)	0.038(2)	0.046(1)	0.029(1)	0.000(1)	0.010(1)	–0.003(1)
C(3)	4*e*	0.4891(3)	–0.3502(2)	0.55144(9)	0.070(2)	0.025(1)	0.030(1)	0.005(1)	0.002(1)	–0.008(1)
C(16)	4*e*	0.9952(3)	0.3171(2)	0.43150(9)	0.056(2)	0.045(1)	0.030(1)	0.000(1)	0.015(1)	0.002(1)
C(15)	4*e*	0.9228(3)	0.3575(2)	0.47415(9)	0.061(2)	0.046(1)	0.037(1)	0.011(1)	0.013(1)	0.004(1)
C(17)	4*e*	1.1102(3)	0.2117(3)	0.4380(1)	0.064(2)	0.069(2)	0.034(1)	0.015(1)	0.023(1)	0.003(1)
C(4)	4*e*	0.4626(3)	–0.2150(2)	0.53576(9)	0.060(2)	0.030(1)	0.027(1)	0.004(1)	0.004(1)	–0.0001(9)
C(10)	4*e*	1.0149(3)	0.2655(2)	0.69875(9)	0.054(2)	0.056(2)	0.032(1)	0.000(1)	0.005(1)	–0.011(1)
C(14)	4*e*	0.9654(3)	0.2958(3)	0.52275(9)	0.061(2)	0.057(2)	0.031(1)	0.004(1)	0.017(1)	–0.005(1)
C(19)	4*e*	0.9497(4)	0.3923(3)	0.37994(9)	0.100(2)	0.061(2)	0.036(2)	0.017(2)	0.029(2)	0.006(1)
C(1)	4*e*	0.5584(3)	–0.5325(2)	0.61914(9)	0.067(2)	0.025(1)	0.035(1)	0.003(1)	0.001(1)	–0.004(1)
C(18)	4*e*	1.1508(3)	0.1483(3)	0.48651(9)	0.052(2)	0.061(2)	0.038(2)	0.015(1)	0.015(1)	0.002(1)
C(22)	4*e*	0.9494(3)	0.1426(2)	0.27525(9)	0.060(2)	0.046(1)	0.034(1)	–0.012(1)	0.007(1)	0.009(1)
C(20)	4*e*	1.0593(3)	0.3417(2)	0.29614(9)	0.058(2)	0.039(1)	0.027(1)	–0.004(1)	0.011(1)	0.006(1)
C(9)	4*e*	0.8947(3)	0.1586(2)	0.63135(9)	0.042(2)	0.046(1)	0.033(1)	0.002(1)	0.005(1)	–0.004(1)
C(21)	4*e*	0.8973(3)	0.1828(3)	0.3209(1)	0.060(2)	0.065(2)	0.042(2)	–0.009(2)	0.017(2)	0.015(1)
C(11)	4*e*	1.1321(3)	0.2356(3)	0.66806(9)	0.041(1)	0.069(2)	0.037(1)	–0.003(1)	0.002(1)	–0.011(1)

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