Crystal structure of monoaqua-(5-bromoisophthalato)-(1,2-bi(4-pyridyl)ethane)zinc(II), $Zn(H_2O)(C_8H_3O_4Br)(C_{12}H_{12}N_2)$

Hui-Ling Li and Yu-Ting Wang*

Henan Institute of Education, Department of Chemistry, Zhengzhou, 450046 Henan, P. R. China

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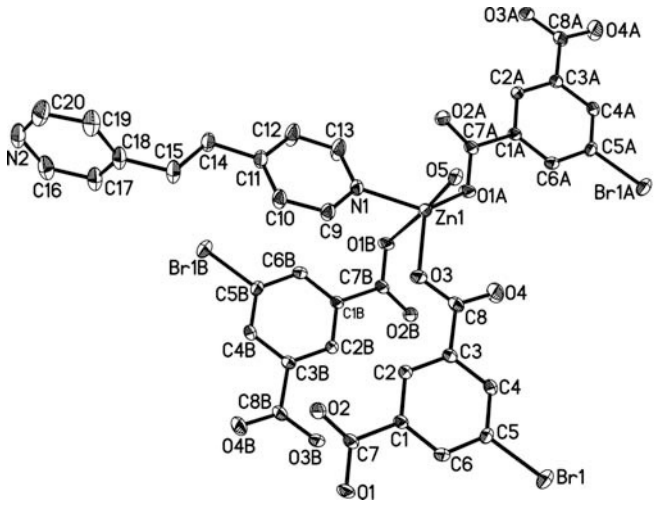

Abstract

$C_{20}H_{17}BrN_2O_5Zn$, triclinic, $P\bar{1}$ (no. 2), a = 8.036(5) Å, b = 9.673(6) Å, c = 14.549(9) Å, α = 105.706(7)°, β = 90.431(7)°, γ = 114.255(7)°, V = 983.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.030, $wR_{ref}(F^2)$ = 0.074, T = 294 K.

Source of material

The title compound was synthesized by the reaction of 1,2-bi(4-pyridyl)ethane (bpa, 0.5 mmol), 5-bromoisophthalic acid (0.5 mmol), $Zn(OAc)_2 \cdot 4H_2O$ (0.5 mmol) and KOH (0.5 mmol) were added to water (12 ml) in a Teflon-lined stainless steel reactor. The mixture was heated at 400 K for 5 d, and then slowly cooled down to room temperature. Colourless crystals of the title compound were obtained.

Discussion

Design of the solid state with molecules that encode well defined noncovalent motifs has recently become a rapidly growing area of research due to the fascinating molecular and/or supramolecular structural diversity [1,2] and potential applications for catalysis [3,4] and material sciences [5,6]. A successful strategy in building such networks is to employ appropriate bridging ligands that can bind metal ions in different modes and provide a possible way to achieve more robust polymeric structures [7,8]. In this context, benzenedicarboxylic acid and its derivatives, such as 1,3-benzenedicarboxylic acid, 1,4-benzenedicarboxylic acid, 5-hydroxyisophthalic acid are widely used as building blocks to link metal ions to produce metal-organic frameworks with interesting structures and properties [9]. Recently, benzenedicarboxylic acid, providing specific rigid molecules, seized our attention for constructing various coordination polymer. The interesting results inspired us to investigate its analogues, 2-bromo-1,4-benzenedicarboxylic acid, for the following reasons: (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 the title crystal structure, the asymmetric unit consists of one zinc(II) ion, one bpa molecule, one 5-bromoisophthalate anion, and one coordinated water molecule. The zinc(II) ion has a distorted trigonal-bipyramidal arrangement (ZnO_4N) with four oxygen atoms from three 5-bromoisophthalate anions and one water molecule, and one nitrogen atom from one bpa molecule. The Zn—N bond lengths are both 2.029(3) Å and the Zn—O bond lengths are in the range of 1.943(2) - 2.823(3) Å, respectively. The bpa molecule adopts an unidentate coordination mode and the unligated N2 atom acts as the hydrogen-bonding acceptor. The 5-bromoisophthalate anion shows μ_3-bridging coordination mode, which link the zinc(II) ions into 1D ladder-like framework. The 5-bromoisophthalate anion, bpa molecule and water molecule are involved in intramolecular O–H···O, and O–H···N hydrogen bonds, which result in a 3D supramolecular structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.06 × 0.18 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	33.15 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7360, 3638
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2975
$N(param)_{refined}$:	262
Programs:	SHELXS-97 [10], SHELXL-97 [11], SHELXTL [12]

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

Atom	Site	x	y	z	U_{iso}
H(1W)	2*i*	1.2344	0.2268	0.3542	0.063
H(2W)	2*i*	1.3422	0.2789	0.4406	0.063
H(2)	2*i*	0.5828	–0.1078	0.4620	0.033
H(4)	2*i*	0.5089	–0.0606	0.2012	0.036
H(6)	2*i*	0.1458	–0.4374	0.2699	0.036
H(9)	2*i*	0.9690	0.1067	0.5896	0.042
H(10)	2*i*	1.0334	0.0893	0.7382	0.046
H(12)	2*i*	1.4039	0.5388	0.8095	0.085
H(13)	2*i*	1.3271	0.5480	0.6615	0.079
H(14A)	2*i*	1.2511	0.3964	0.9394	0.046
H(14B)	2*i*	1.4065	0.3454	0.9040	0.046

* Correspondence author (e-mail: hnjyxywyt@126.com)

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(15A)	2*i*	1.0429	0.1184	0.8959	0.056
H(15B)	2*i*	1.2149	0.0816	0.8791	0.056
H(16)	2*i*	1.0555	0.2744	1.2222	0.065
H(17)	2*i*	1.014	0.2562	1.0632	0.061
H(19)	2*i*	1.4135	0.1095	1.0207	0.067
H(20)	2*i*	1.4482	0.1391	1.1822	0.072

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(1)	2*i*	1.06952(4)	0.32842(4)	0.47614(2)	0.0310(2)	0.0299(2)	0.0296(2)	–0.0042(1)	–0.0043(1)	0.0183(2)
Br(1)	2*i*	0.18433(5)	–0.33982(4)	0.09478(2)	0.0489(2)	0.0543(2)	0.0293(2)	0.0141(2)	–0.0081(1)	0.0093(2)
O(1)	2*i*	0.1294(3)	–0.4920(2)	0.4264(2)	0.026(1)	0.036(1)	0.050(1)	–0.0020(9)	–0.0013(9)	0.029(1)
O(2)	2*i*	0.4082(3)	–0.3675(3)	0.5116(2)	0.033(1)	0.050(1)	0.044(1)	0.005(1)	–0.003(1)	0.032(1)
O(3)	2*i*	0.8316(3)	0.1439(2)	0.4439(2)	0.030(1)	0.029(1)	0.040(1)	–0.0026(9)	–0.0012(9)	0.0146(9)
O(4)	2*i*	0.7862(4)	0.1661(3)	0.2987(2)	0.071(2)	0.047(1)	0.046(2)	–0.013(1)	0.003(1)	0.028(1)
O(5)	2*i*	1.2363(3)	0.2280(3)	0.4108(1)	0.038(1)	0.051(1)	0.028(1)	0.008(1)	–0.0019(9)	0.016(1)
N(1)	2*i*	1.1394(3)	0.3288(3)	0.6105(2)	0.036(1)	0.033(1)	0.026(1)	0.007(1)	0.003(1)	0.015(1)
N(2)	2*i*	1.2545(5)	0.2063(3)	1.2179(2)	0.085(2)	0.039(2)	0.031(2)	0.010(2)	–0.002(2)	0.016(1)
C(1)	2*i*	0.3564(4)	–0.2884(3)	0.3761(2)	0.023(1)	0.022(1)	0.029(1)	0.005(1)	0.003(1)	0.012(1)
C(2)	2*i*	0.5145(4)	–0.1452(3)	0.4015(2)	0.026(1)	0.027(1)	0.026(1)	0.006(1)	0.002(1)	0.011(1)
C(3)	2*i*	0.5702(4)	–0.0585(3)	0.3368(2)	0.027(1)	0.022(1)	0.030(2)	0.005(1)	0.004(1)	0.010(1)
C(4)	2*i*	0.4709(4)	–0.1167(3)	0.2457(2)	0.031(2)	0.030(2)	0.030(2)	0.010(1)	0.008(1)	0.016(1)
C(5)	2*i*	0.3155(4)	–0.2587(3)	0.2221(2)	0.033(2)	0.032(2)	0.021(1)	0.011(1)	–0.001(1)	0.007(1)
C(6)	2*i*	0.2541(4)	–0.3440(3)	0.2865(2)	0.024(1)	0.026(1)	0.033(2)	0.003(1)	0.001(1)	0.009(1)
C(7)	2*i*	0.2980(4)	–0.3872(3)	0.4450(2)	0.028(2)	0.027(1)	0.035(2)	0.006(1)	0.006(1)	0.016(1)
C(8)	2*i*	0.7418(4)	0.0968(3)	0.3599(2)	0.034(2)	0.028(2)	0.037(2)	0.004(1)	0.008(1)	0.016(1)
C(9)	2*i*	1.0569(4)	0.1961(3)	0.6350(2)	0.043(2)	0.029(2)	0.029(2)	0.008(1)	–0.000(1)	0.012(1)
C(10)	2*i*	1.0955(4)	0.1847(4)	0.7244(2)	0.051(2)	0.032(2)	0.031(2)	0.014(1)	0.005(1)	0.017(1)
C(11)	2*i*	1.2249(4)	0.3131(4)	0.7930(2)	0.042(2)	0.039(2)	0.026(2)	0.015(1)	0.005(1)	0.014(1)
C(12)	2*i*	1.3124(6)	0.4489(4)	0.7662(2)	0.086(3)	0.045(2)	0.035(2)	–0.021(2)	–0.020(2)	0.020(2)
C(13)	2*i*	1.2667(6)	0.4537(4)	0.6768(2)	0.082(3)	0.041(2)	0.039(2)	–0.016(2)	–0.015(2)	0.025(2)
C(14)	2*i*	1.2753(5)	0.3151(4)	0.8938(2)	0.048(2)	0.040(2)	0.025(2)	0.014(2)	0.002(1)	0.013(1)
C(15)	2*i*	1.1739(5)	0.1587(4)	0.9159(2)	0.070(2)	0.042(2)	0.027(2)	0.021(2)	0.007(2)	0.015(1)
C(16)	2*i*	1.1286(6)	0.2409(4)	1.1811(2)	0.083(3)	0.039(2)	0.033(2)	0.017(2)	0.014(2)	0.012(2)
C(17)	2*i*	1.1020(5)	0.2292(4)	1.0852(2)	0.074(3)	0.048(2)	0.036(2)	0.026(2)	0.010(2)	0.022(2)
C(18)	2*i*	1.2061(5)	0.1772(4)	1.0214(2)	0.068(2)	0.035(2)	0.028(2)	0.019(2)	0.007(2)	0.015(1)
C(19)	2*i*	1.3384(6)	0.1434(4)	1.0601(3)	0.082(3)	0.055(2)	0.043(2)	0.035(2)	0.012(2)	0.023(2)
C(20)	2*i*	1.3576(6)	0.1607(4)	1.1575(3)	0.085(3)	0.052(2)	0.047(2)	0.031(2)	–0.005(2)	0.022(2)

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