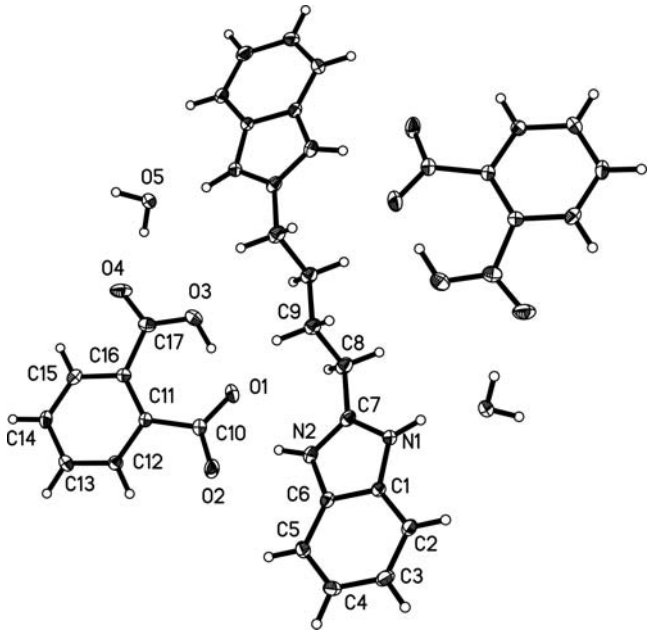
Crystal structure of 2,2′-(1,4-butanediyl)bis(3*H*-benzimidazolium) bis(2-carboxybenzoate) dihydrate, $[C_{18}H_{20}N_4][C_8H_5O_4]_2 \cdot 2H_2O$

Pu-Hong Wen*

Baoji University of Arts and Science, Department of Chemistry and Chemical Engineering, Baoji 721013, Shaanxi, P. R. China

Received December 9, 2010, accepted and available on-line January 25, 2011; CCDC no. 1267/3326



Abstract

$C_{34}H_{34}N_4O_{10}$, monoclinic, *C*12/*c*1 (no. 15), a = 16.538(5) Å, b = 14.588(4) Å, c = 14.760(4) Å, β = 118.765(4)°, V = 3121.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.111, T = 143 K.

Source of material

A mixture of 2,2′-(1,4-butanediyl)bis(3*H*-benzimidazole) (87.0 mg, 0.3 mmol), phthalic acid (99.6 mg, 0.6 mmol), and H_2O (14 mL) was sealed in a 25 mL Teflon-lined stainless steel container, which was heated at 423 K for 72 h. Some colourless block-like crystals were obtained after cooling down to room temperature at a rate of 5 K/h. Single crystals suitable for X-ray analysis were isolated.

Experimental details

All hydrogen atoms bound to carbon atoms were placed in calculated positions and refined in a riding model with d(C—H) = 0.95 Å (Csp^2) or 0.99 Å (Csp^3), and U_{iso}(H) = 1.2 U_{eq}(C). Hydrogen atoms bound to nitrogen and oxygen atoms were found in the difference Fourier maps and refined freely.

Discussion

In the past decades, much attention has been focused on the coordination polymers because of their potential applications in microelectronics, nonlinear optics or as fluorescent, porous materials and catalysis [1]. Most coordination polymers are constructed by using appropriate organic ligands, especially bridging ligands containing oxygen and nitrogen, to coordinate to metal centers [2]. Bis(2-benzimidazole) and some substituted bis(2-benzimidazolyl)alkanes are attractive choice for this purpose because of their multifunctional linking groups [3]. They are known to be strong chelating agents coordinating through both nitrogen atoms [4]. A number of cases were reported in which a proton transferred from a carboxylic acid to an amine formed novel proton transfer compounds [5].

The crystal structure of the title compound shows two proton from two phthalic acid molecules transferred to the N atoms of bis-benzimidazole. There are three types of hydrogen bonds in the crystal structure. The intramolecular hydrogen bond (O3–H3O···O1) is formed by the carboxyl and the carboxylate group in 2-carboxybenzoate. Further intermolecular hydrogen bonds are formed by water molecules with 2,2′-(1,4-butanediyl)bis(3*H*-benzimidazolium) (N1–H1N···O5) and 2-carboxybenzoate anions (O5–H5A···O4 and O5–H5B···O2). The two latter cause to generate a lepidocrocite-like layered structure. These layeres are linked together mostly by π-π stacking interaction of aromatic nucleus.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.25 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.05 cm^{-1}
Diffractometer, scan mode:	Rigaku, AFC10/Saturn724+, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13715, 3568
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2704
$N(param)_{refined}$:	237
Programs:	SHELXS-97, SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(2)	8*f*	0.5273	0.9539	0.6186	0.034
H(3)	8*f*	0.6495	1.0218	0.7642	0.041
H(4)	8*f*	0.7528	0.9328	0.9022	0.044
H(5)	8*f*	0.7398	0.7730	0.9017	0.039
H(8A)	8*f*	0.4125	0.6090	0.5257	0.041
H(8B)	8*f*	0.4701	0.5494	0.6279	0.041
H(9A)	8*f*	0.5288	0.5856	0.4767	0.035
H(9B)	8*f*	0.5889	0.5283	0.5804	0.035
H(12)	8*f*	0.1585	0.4881	0.4722	0.030
H(13)	8*f*	0.0488	0.3823	0.3664	0.034
H(14)	8*f*	0.0621	0.2289	0.4188	0.035

* e-mail: wenpuhong@gmail.com

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15)	8*f*	0.1849	0.1846	0.5755	0.032
H(5A)	8*f*	0.133(2)	0.662(2)	0.628(2)	0.075(7)
H(1N)	8*f*	0.454(1)	0.781(1)	0.539(1)	0.043(5)
H(5B)	8*f*	0.172(2)	0.615(2)	0.573(2)	0.072(7)
H(2N)	8*f*	0.628(1)	0.628(1)	0.769(2)	0.060(6)
H(3O)	8*f*	0.392(2)	0.375(2)	0.760(2)	0.087(8)

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}
O(1)	8*f*	0.38327(8)	0.46449(9)	0.7226(1)	0.0324(6)	0.0467(7)	0.0500(7)	–0.0146(5)	–0.0058(5)	0.0076(6)
O(2)	8*f*	0.28234(7)	0.55943(6)	0.61210(9)	0.0313(6)	0.0229(5)	0.0461(7)	–0.0035(4)	0.0128(5)	–0.0073(4)
O(3)	8*f*	0.39352(8)	0.3097(1)	0.78172(9)	0.0315(6)	0.0509(8)	0.0458(7)	–0.0006(6)	–0.0008(5)	0.0182(6)
O(4)	8*f*	0.31149(9)	0.18429(7)	0.73594(8)	0.0654(8)	0.0273(5)	0.0316(6)	0.0104(5)	0.0200(6)	0.0073(4)
O(5)	8*f*	0.11817(8)	0.64627(8)	0.56162(9)	0.0374(6)	0.0404(6)	0.0256(6)	0.0134(5)	0.0039(5)	–0.0034(5)
N(1)	8*f*	0.50023(8)	0.76257(8)	0.60250(9)	0.0219(6)	0.0241(6)	0.0246(6)	0.0018(5)	0.0091(5)	–0.0015(5)
N(2)	8*f*	0.59832(8)	0.67897(8)	0.73045(9)	0.0294(6)	0.0212(6)	0.0331(7)	0.0043(5)	0.0156(5)	0.0043(5)
C(1)	8*f*	0.56248(9)	0.82293(9)	0.6757(1)	0.0222(6)	0.0242(6)	0.0229(7)	0.0032(5)	0.0115(5)	–0.0013(5)
C(2)	8*f*	0.5698(1)	0.91787(9)	0.6752(1)	0.0317(7)	0.0230(7)	0.0305(7)	0.0056(6)	0.0149(6)	0.0007(6)
C(3)	8*f*	0.6419(1)	0.9571(1)	0.7612(1)	0.0378(8)	0.0246(7)	0.0412(9)	0.0001(6)	0.0199(7)	–0.0084(6)
C(4)	8*f*	0.7042(1)	0.9033(1)	0.8443(1)	0.0301(8)	0.0386(8)	0.0336(8)	0.0001(7)	0.0093(7)	–0.0145(7)
C(5)	8*f*	0.6974(1)	0.8089(1)	0.8450(1)	0.0287(7)	0.0385(8)	0.0238(7)	0.0089(6)	0.0073(6)	–0.0009(6)
C(6)	8*f*	0.62478(9)	0.76943(9)	0.7577(1)	0.0269(7)	0.0232(6)	0.0270(7)	0.0046(5)	0.0153(6)	0.0013(5)
C(7)	8*f*	0.52401(9)	0.67697(9)	0.6368(1)	0.0243(7)	0.0235(7)	0.0371(8)	0.0001(5)	0.0194(6)	–0.0022(6)
C(8)	8*f*	0.4756(1)	0.5924(1)	0.5793(1)	0.0269(7)	0.0257(7)	0.0492(9)	–0.0044(6)	0.0187(7)	–0.0081(6)
C(9)	8*f*	0.5252(1)	0.54369(9)	0.5274(1)	0.0280(7)	0.0226(7)	0.0388(8)	–0.0043(6)	0.0171(6)	–0.0031(6)
C(10)	8*f*	0.3055(1)	0.47976(9)	0.6450(1)	0.0251(7)	0.0282(7)	0.0291(7)	–0.0034(6)	0.0126(6)	–0.0036(6)
C(11)	8*f*	0.24022(9)	0.40142(9)	0.5879(1)	0.0213(6)	0.0222(6)	0.0235(7)	–0.0008(5)	0.0121(5)	–0.0023(5)
C(12)	8*f*	0.16483(9)	0.42591(9)	0.4938(1)	0.0265(7)	0.0229(6)	0.0253(7)	0.0003(5)	0.0115(6)	–0.0002(5)
C(13)	8*f*	0.0989(1)	0.3633(1)	0.4305(1)	0.0238(7)	0.0342(8)	0.0233(7)	–0.0004(6)	0.0076(6)	–0.0025(6)
C(14)	8*f*	0.1067(1)	0.2728(1)	0.4613(1)	0.0278(7)	0.0302(7)	0.0302(7)	–0.0102(6)	0.0148(6)	–0.0093(6)
C(15)	8*f*	0.1802(1)	0.24688(9)	0.5547(1)	0.0325(8)	0.0223(6)	0.0320(7)	–0.0025(6)	0.0209(6)	–0.0017(6)
C(16)	8*f*	0.24785(9)	0.30854(9)	0.6197(1)	0.0236(7)	0.0244(6)	0.0236(7)	0.0025(5)	0.0135(6)	0.0008(5)
C(17)	8*f*	0.3217(1)	0.2641(1)	0.7180(1)	0.0323(8)	0.0319(8)	0.0289(7)	0.0075(6)	0.0164(6)	0.0038(6)

Acknowledgments. This work was supported by Science and Technology Foundation of the Shaanxi Key Laboratory (grant no. 2003JS018), Scientific Research Project (grant no. 09JK334) from Shaanxi Province Office of Education and Key Research Project (grant nos. ZK1051, ZK1035) from Baoji University of Arts and Sciences.

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