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3-(Benzimidazolium-2-yl)propionate dihydrate

Xin Xiao,[a] Yun-Qiang Zhang,[a] Sai-Feng Xue[a] and Zhu Tao[b]*

[a]Key Laboratory of Macrocyclic and Supramolecular Chemistry of Guizhou Province, Guizhou University, Guiyang 550025, People's Republic of China, and [b]Institute of Applied Chemistry, Guizhou University, Guiyang 550025, People's Republic of China
Correspondence e-mail: gyhxxiaoxin@163.com

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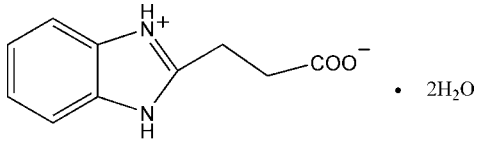
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.113; data-to-parameter ratio = 12.2.

In the crystal struture of the title compound, $C_{10}H_{10}N_2O_2\cdot 2H_2O$, the component species are linked to the water molecules by N—H···O and O—H···O hydrogen bonds to form a three-dimensional network structure.

Related literature

For general background, see: Day & Arnold (2000); Day *et al.* (2002); Freeman *et al.* (1981); Kim *et al.* (2000). For related structure, see: Ge *et al.* (2007).



Experimental

Crystal data

$C_{10}H_{10}N_2O_2\cdot 2H_2O$
M_r = 226.23
Monoclinic, $P2_1/c$
a = 18.444 (3) Å
b = 4.9730 (8) Å
c = 11.9097 (19) Å
β = 94.530 (5)°
V = 1089.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 273 (2) K
0.29 × 0.26 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: none
7787 measured reflections
2002 independent reflections
1394 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.113
S = 1.08
1971 reflections
161 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.86	1.86	2.700 (2)	166
N2—H2N···O2^{ii}	0.86	1.80	2.654 (3)	170
O2W—H2W1···O1W^{iii}	0.841 (17)	2.007 (18)	2.842 (3)	172 (3)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2490).

References

Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Day, A. I. & Arnold, A. P. (2000). Int. Patent WO/2000/068232.
Day, A. I., Blanch, R. J., Arnold, A. P., Lorenzo, S., Lewis, G. R. & Dance, I. (2002). *Angew. Chem. Int. Ed.* **41**, 275–277.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Freeman, W. A., Mock, W. L. & Shih, N. Y. (1981). *J. Am. Chem. Soc.* **103**, 7367–7368.
Ge, J. Y., Xue, S. F., Zhu, Q. J., Tao, Z. & Zhang, J., X. (2007). *J. Incl. Phenom. Macro.* **58**, 63–69.
Kim, J., Jung, I.-S., Kim, S.-Y., Lee, E., Kang, J.-K., Sakamoto, S., Yamaguchi, K. & Kim, K. (2000). *J. Am. Chem. Soc.* **122**, 540–541.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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3-(Benzimidazolium-2-yl)propionate dihydrate

Xin Xiao, Yun-Qiang Zhang, Sai-Feng Xue and Zhu Tao

S1. Comment

As part of our ongoing investigation on benzimidazole compounds, we present a compound containing multiple functional groups that can develop strong intermolecular interactions with cucurbit[*n*]urils (CB[*n*]) (Freeman *et al.*, 1981; Day & Arnold, 2000; Day *et al.*, 2002; Kim *et al.*, 2000; Ge *et al.*, 2007).

The crystal structure of the title compound (Fig. 1) consists of a 3-(1*H*-benzo[*d*]imidazol-2-yl) propanoic acid organic molecule and two lattice water molecules. the dihedral angle between the benzene ring (C1,C2,C3,C4,C5,C6) and the imidazole ring (C5,C6,C9,N2,C7,N1) is 0.61 (13)°. The C7—C8—C9—C10 torsion angle is -66.3 (3)°. The title compound forms intermolecular H bonds whereas the protonated N1 and N2 atoms act as hydrogen-bond donors and the O1 and O2 atoms act as hydrogen-bond acceptors, the O—H···O hydrogen bonds are also observed between the water molecules O2W and O1W (Table 1). these contacts and the cross-linking interactions stabilize the crystal packing.

S2. Experimental

The propionic anhydride (13 g, 0.1 mol) was dissolved in hot water (100 ml) with stirring, and a warm solution of 1,2-di-aminobenzene(10.8 g, 0.1 mol) in 1,4-dioxane (100 ml) was added, following by the addition of ployphosphoric acid (50 ml) as catalyst. The mixture was refluxed for 8 h and then cooled, the solution was filtered and the filtrate was set aside for three weeks to obtain colorless crystals.

S3. Refinement

Water H atoms were located in a difference Fourier map and refined as riding in their as-found positions relative to O atoms with U_{iso}(H) = 1.2U_{eq}(O). All other H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.97 Å, N—H = 0.86 Å, and U_{iso}(H) = 1.2–1.5 U_{eq}(C,*N*).

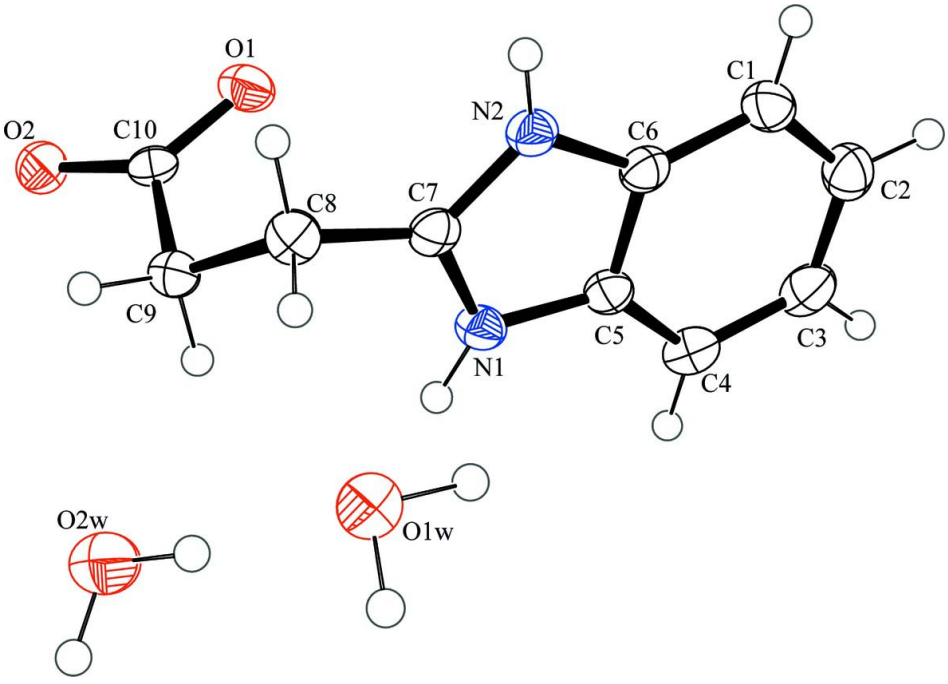

Figure 1

The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

$C_{10}H_{10}N_2O_2\cdot 2H_2O$
$M_r = 226.23$
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Hall symbol: -P 2ybc
$a = 18.444\ (3)$ Å
$b = 4.9730\ (8)$ Å
$c = 11.9097\ (19)$ Å
$\beta = 94.530\ (5)°$
$V = 1089.0\ (3)$ Å^3
$Z = 4$

$F(000) = 480$
$D_x = 1.380$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2002 reflections
$\theta = 1.1–25.4°$
$\mu = 0.11$ mm^{-1}
$T = 273$ K
Prism, colourless
0.29 × 0.26 × 0.20 mm

Data collection

Bruker CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
7787 measured reflections
2002 independent reflections

1394 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.1°$
$h = -21\rightarrow19$
$k = -5\rightarrow4$
$l = -13\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.113$
$S = 1.08$
1971 reflections
161 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.5171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.24941 (9)	0.3021 (3)	0.56604 (13)	0.0306 (5)
O2	0.31408 (9)	0.3485 (3)	0.41718 (12)	0.0285 (4)
O1W	0.45721 (11)	0.3587 (4)	0.36594 (15)	0.0360 (5)
O2W	0.52898 (12)	−0.1307 (4)	0.37002 (17)	0.0398 (5)
N1	0.22255 (10)	0.8913 (4)	0.70511 (15)	0.0249 (5)
H1N	0.2296	1.0021	0.6516	0.030*
N2	0.23986 (10)	0.5625 (4)	0.82389 (15)	0.0241 (5)
H2N	0.2595	0.4266	0.8591	0.029*
C1	0.12302 (14)	0.6171 (5)	0.92071 (19)	0.0290 (6)
H1A	0.1303	0.4775	0.9723	0.035*
C2	0.06132 (14)	0.7751 (5)	0.9168 (2)	0.0331 (7)
H2A	0.0264	0.7417	0.9673	0.040*
C3	0.04988 (14)	0.9848 (5)	0.8385 (2)	0.0334 (7)
H3A	0.0074	1.0858	0.8380	0.040*
C4	0.09993 (13)	1.0447 (5)	0.7624 (2)	0.0300 (6)
H4A	0.0924	1.1838	0.7107	0.036*
C5	0.16226 (13)	0.8870 (5)	0.76689 (18)	0.0237 (6)
C6	0.17353 (13)	0.6765 (5)	0.84417 (18)	0.0238 (6)
C7	0.26826 (13)	0.6960 (5)	0.74162 (18)	0.0234 (6)
C8	0.34047 (13)	0.6448 (5)	0.69974 (18)	0.0264 (6)
H8A	0.3582	0.4724	0.7285	0.032*
H8B	0.3741	0.7816	0.7299	0.032*
C9	0.34097 (13)	0.6433 (5)	0.57180 (18)	0.0252 (6)
H9A	0.3207	0.8115	0.5425	0.030*

H9B	0.3909	0.6334	0.5521	0.030*
C10	0.29840 (13)	0.4120 (5)	0.51556 (18)	0.0227 (6)
H1W1	0.4133 (12)	0.352 (7)	0.386 (3)	0.084 (13)*
H1W2	0.4755 (16)	0.208 (4)	0.384 (3)	0.059 (11)*
H2W1	0.5341 (16)	−0.149 (6)	0.3009 (16)	0.053 (9)*
H2W2	0.5073 (19)	−0.268 (5)	0.387 (3)	0.089 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0340 (10)	0.0248 (10)	0.0334 (9)	−0.0060 (8)	0.0054 (8)	0.0027 (8)
O2	0.0334 (10)	0.0268 (10)	0.0255 (8)	−0.0038 (8)	0.0042 (8)	−0.0056 (7)
O1W	0.0374 (13)	0.0303 (12)	0.0416 (11)	−0.0013 (10)	0.0106 (10)	−0.0001 (9)
O2W	0.0486 (13)	0.0315 (12)	0.0399 (11)	−0.0038 (11)	0.0079 (10)	−0.0010 (10)
N1	0.0311 (12)	0.0209 (12)	0.0230 (9)	−0.0003 (10)	0.0032 (9)	0.0034 (9)
N2	0.0290 (12)	0.0205 (12)	0.0225 (9)	0.0022 (10)	0.0013 (9)	0.0013 (8)
C1	0.0336 (15)	0.0257 (15)	0.0281 (12)	−0.0059 (13)	0.0043 (11)	−0.0011 (11)
C2	0.0315 (15)	0.0340 (16)	0.0348 (14)	−0.0053 (13)	0.0090 (12)	−0.0058 (12)
C3	0.0292 (15)	0.0272 (15)	0.0438 (15)	0.0027 (13)	0.0035 (13)	−0.0062 (13)
C4	0.0309 (15)	0.0231 (15)	0.0354 (13)	0.0009 (12)	−0.0006 (12)	−0.0010 (11)
C5	0.0267 (14)	0.0194 (14)	0.0248 (11)	−0.0044 (11)	0.0016 (11)	−0.0032 (10)
C6	0.0268 (14)	0.0198 (13)	0.0248 (12)	−0.0005 (11)	0.0010 (11)	−0.0029 (10)
C7	0.0289 (14)	0.0202 (13)	0.0206 (11)	−0.0035 (12)	−0.0004 (10)	−0.0045 (10)
C8	0.0240 (14)	0.0271 (15)	0.0279 (12)	−0.0009 (12)	0.0006 (11)	−0.0011 (11)
C9	0.0289 (14)	0.0208 (14)	0.0264 (12)	−0.0028 (12)	0.0049 (11)	0.0005 (11)
C10	0.0257 (14)	0.0167 (13)	0.0256 (12)	0.0048 (11)	0.0012 (11)	0.0050 (10)

Geometric parameters (Å, º)

O1—C10	1.250 (3)	C2—C3	1.404 (4)
O2—C10	1.269 (3)	C2—H2A	0.9300
O1W—H1W1	0.863 (18)	C3—C4	1.376 (3)
O1W—H1W2	0.845 (18)	C3—H3A	0.9300
O2W—H2W1	0.841 (17)	C4—C5	1.389 (3)
O2W—H2W2	0.823 (18)	C4—H4A	0.9300
N1—C7	1.336 (3)	C5—C6	1.399 (3)
N1—C5	1.381 (3)	C7—C8	1.481 (3)
N1—H1N	0.8600	C8—C9	1.525 (3)
N2—C7	1.325 (3)	C8—H8A	0.9700
N2—C6	1.387 (3)	C8—H8B	0.9700
N2—H2N	0.8600	C9—C10	1.518 (3)
C1—C2	1.380 (3)	C9—H9A	0.9700
C1—C6	1.386 (3)	C9—H9B	0.9700
C1—H1A	0.9300		
H1W1—O1W—H1W2	105 (3)	C4—C5—C6	121.8 (2)
H2W1—O2W—H2W2	104 (3)	C1—C6—N2	132.5 (2)
C7—N1—C5	109.22 (19)	C1—C6—C5	121.3 (2)

C7—N1—H1N	125.4	N2—C6—C5	106.2 (2)
C5—N1—H1N	125.4	N2—C7—N1	109.2 (2)
C7—N2—C6	109.2 (2)	N2—C7—C8	125.6 (2)
C7—N2—H2N	125.4	N1—C7—C8	125.2 (2)
C6—N2—H2N	125.4	C7—C8—C9	114.5 (2)
C2—C1—C6	116.9 (2)	C7—C8—H8A	108.6
C2—C1—H1A	121.6	C9—C8—H8A	108.6
C6—C1—H1A	121.6	C7—C8—H8B	108.6
C1—C2—C3	121.7 (2)	C9—C8—H8B	108.6
C1—C2—H2A	119.2	H8A—C8—H8B	107.6
C3—C2—H2A	119.2	C10—C9—C8	113.63 (19)
C4—C3—C2	121.6 (2)	C10—C9—H9A	108.8
C4—C3—H3A	119.2	C8—C9—H9A	108.8
C2—C3—H3A	119.2	C10—C9—H9B	108.8
C3—C4—C5	116.7 (2)	C8—C9—H9B	108.8
C3—C4—H4A	121.7	H9A—C9—H9B	107.7
C5—C4—H4A	121.7	O1—C10—O2	124.2 (2)
N1—C5—C4	132.0 (2)	O1—C10—C9	119.2 (2)
N1—C5—C6	106.1 (2)	O2—C10—C9	116.6 (2)
C6—C1—C2—C3	0.5 (4)	C4—C5—C6—C1	−0.5 (3)
C1—C2—C3—C4	−0.6 (4)	N1—C5—C6—N2	−0.7 (2)
C2—C3—C4—C5	0.1 (4)	C4—C5—C6—N2	179.2 (2)
C7—N1—C5—C4	−179.9 (2)	C6—N2—C7—N1	−1.2 (3)
C7—N1—C5—C6	0.0 (2)	C6—N2—C7—C8	176.5 (2)
C3—C4—C5—N1	−179.6 (2)	C5—N1—C7—N2	0.7 (2)
C3—C4—C5—C6	0.4 (3)	C5—N1—C7—C8	−177.0 (2)
C2—C1—C6—N2	−179.6 (2)	N2—C7—C8—C9	135.7 (2)
C2—C1—C6—C5	0.1 (3)	N1—C7—C8—C9	−47.0 (3)
C7—N2—C6—C1	−179.1 (2)	C7—C8—C9—C10	−66.3 (3)
C7—N2—C6—C5	1.2 (2)	C8—C9—C10—O1	23.1 (3)
N1—C5—C6—C1	179.5 (2)	C8—C9—C10—O2	−159.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.86	1.86	2.700 (2)	166
N2—H2*N*···O2^{ii}	0.86	1.80	2.654 (3)	170
O2*W*—H2*W*1···O1*W*^{iii}	0.84 (2)	2.01 (2)	2.842 (3)	172 (3)

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