Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Ethyl-1*H*-imidazole-4-carboxylate monohydrate

Shi-Jie Li,[a] Juan-Hua Liu,[a] Wen-Dong Song,[b]* Xiao-Fei Li[c] and Dong-Liang Miao[a]

[a]College of Food Science and Technology, Guangdong Ocean University, Zhanjiang 524088, People's Republic of China, [b]College of Science, Guangdong Ocean University, Zhanjiang 524088, People's Republic of China, and [c]College of Agriculture, Guangdong Ocean University, Zhanjiang 524088, People's Republic of China
Correspondence e-mail: songwd60@163.com

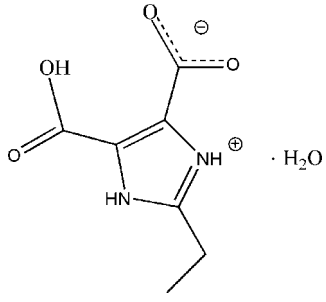
Received 9 March 2011; accepted 23 March 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.106; data-to-parameter ratio = 11.7.

In the title compound, $C_7H_8N_2O_4\cdot H_2O$, the imidazole N atom is protonated and one of the carboxylate groups is deprotonated, forming a zwitterion. The two carboxyl groups are are approximately coplanar with the imidazole ring [O—C—C—C torsion angles = −176.8 (2) and 2.9 (4)° for one group and −4.6 (3) and 176.4 (2)° for the other] and have an intramolecular O—H···O hydrogen bond between them. The water molecule is linked to the organic molecules *via* an N—H···O hydrogen bonds. Intermolecular O—H···O and N—H···O hydrogen bonds are found in the crystal structure.

Related literature

For our past work based on the 2-propyl-1H-imidazole-4,5-carboxylate (H_3pimda) ligand, see: Yan *et al.* (2010); Li *et al.* (2010); Song *et al.* (2010); He *et al.* (2010); Fan *et al.* (2010). For related coordination polymers based on H_3EIDC (2-ethyl-1H-imidazole-4,5-dicarboxylate), see: Wang *et al.* (2008); Zhang *et al.* (2010); Li *et al.* (2011). For the synthesis of H_3EIDC, see: Sun *et al.* (2006).



Experimental

Crystal data

$C_7H_{10}N_2O_5$
M_r = 202.17
Monoclinic, $P2_1/c$
a = 7.6132 (6) Å
b = 14.3779 (16) Å
c = 7.9396 (8) Å
β = 97.799 (1)°
V = 861.04 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 K
0.50 × 0.41 × 0.40 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.936, T_{max} = 0.948
4224 measured reflections
1510 independent reflections
1166 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.05
1510 reflections
129 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.86	1.91	2.754 (2)	168
N2—H2···O1W^{ii}	0.86	1.89	2.751 (2)	177
O2—H2A···O3	0.82	1.63	2.452 (2)	176
O1W—H1W···O2^{iii}	0.85	2.05	2.8863 (19)	169
O1W—H2W···O1	0.85	2.03	2.849 (2)	163

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The work was supported by the Nonprofit Industry Foundation of the National Ocean Administration of China (grant No. 2000905021), the Guangdong Ocean Fisheries Technology Promotion Project [grant No. A2009003–018(c)], the Guangdong Chinese Academy of Science Comprehensive Strategic Cooperation Project (grant No. 2009B091300121), the Guangdong Province Key Project in the Field of Social Development [grant No. A2009011–007(c)], the Science and Technology Department of Guangdong Province Project (grant No. 00087 061110314018) and the Guangdong Natural Science Foundation (No. 9252408801000002).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2272).

References

Bruker (2007). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fan, R.-Z., Li, S.-J., Song, W.-D., Miao, D.-L. & Hu, S.-W. (2010). *Acta Cryst.* E**66**, m897–m898.

He, L.-Z., Li, S.-J., Song, W.-D. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m896.

Li, S.-J., Ma, X.-T., Song, W.-D., Li, X.-F. & Liu, J.-H. (2011). *Acta Cryst.* E**67**, m295–m296.

Li, S.-J., Yan, J.-B., Song, W.-D., Wang, H. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m280.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Song, W.-D., Yan, J.-B., Li, S.-J., Miao, D.-L. & Li, X.-F. (2010). *Acta Cryst.* E**66**, m53.

Sun, T., Ma, J.-P., Huang, R.-Q. & Dong, Y.-B. (2006). *Acta Cryst.* E**62**, o2751–o2752.

Wang, S., Zhang, L. R., Li, G. H., Huo, Q. S. & Liu, Y. L. (2008). *CrystEngComm* **10**, 1662–1666.

Yan, J.-B., Li, S.-J., Song, W.-D., Wang, H. & Miao, D.-L. (2010). *Acta Cryst.* E**66**, m99.

Zhang, F. W., Li, Z. F., Ge, T. Z., Yao, H. C., Li, G., Lu, H. J. & Zhu, Y. Y. (2010). *Inorg. Chem.* **49**, 3776–3788.

supplementary materials

Acta Cryst. (2011). E**67**, o996-o997 [**doi:10.1107/S1600536811010774**]

2-Ethyl-1*H*-imidazole-4-carboxylate monohydrate

S.-J. Li, J.-H. Liu, W.-D. Song, X.-F. Li and D.-L. Miao

Comment

4,5-imidazoledicarboxylic acid (H_3IDC) ligand posesses great potential for coordination interactions and hydrogen bonding, can be deprotonated to generate H_2IDC^-, $HIDC_2^-$ and IDC_3^- anions at different pH values. Up to date, it has been widely studied. 2-propyl-1H-imidazole-4,5-carboxylate (H_3pimda) ligand as one derivative of H_3IDC with efficient N,O-donors has been used to obtain new metal-organic complexes by our research group(Song *et al.*, 2010; Yan *et al.*, 2010; He *et al.* 2010; Fan *et al.* 2010; Li *et al.* 2010). Recently, an analogue of H_3IDC, 2-ethyl-1H-imidazole-4,5-dicarboxylate (H_3EIDC)ligand has also been used to construct intriguing coordination polymers (Wang *et al.*, 2008; Zhang *et al.* , 2010; Li *et al.*, 2011;). However, the crystal structure of H_3EIDC ligand has not been determined. Considering that in mind, we focus on obtaining the crystal and its crystal structure will be reported here.

As illustrated in Fig. 1, the title compound, $(C_7H_8N_2O_4).H_2O$, crystallizes as a zwitterion in which the imidazole N atom is protonated, one of the carboxylate groups is deprontonated. The two carboxyl groups are approximately coplanar with the imidazole ring, as indicated by the fact that the O1—C1—C2—C3 and O2—C1—C2—C3 torsion angles are -176.8 (2) ° and 2.9 (4) °, respectively; the O3—C4—C3—C2 and O4—C4—C3—C2 torsion angles are -4.6 (3) ° and 176.4 (2) °, respectively. The solvent water molecules are linked to the organic ligands via N—H···O and O—H···O hydrogen bonds(Table 1), which stabilize the three-dimensional network(Fig. 2).

Experimental

The organic molecule powder was abtained from 2-ethylbenzimidazole according to a literature procedure (Sun *et al.* 2006), then the 2-ethyl-1H-imidazole-4,5-dicarboxylate(0.5 mmol, 0.9 g) was disolved in 15 ml of H_2O solution with the pH of 6 adjusted by NaOH, colorless crystals was obtained by slow evaporation of the solvent at room temperature.

Refinement

H atoms of the water molecule were located in a difference Fourier map and refined as riding with an O—H distance restraint of 0.84 (1) Å, with U_{iso}(H) = 1.5 U_{eq}. The H···H distances within the water molecules were restraint to 1.39 (1) Å. Carboxyl H atoms were located in a difference map but were refined as riding on the parent O atoms with with O—H = 0.82 Å with U_{iso}(H) = 1.5 U_{eq}(O). Carbon and nitrogen bound H atoms were placed at calculated positions and were treated as riding on the parent C or N atoms with C—H = 0.96 (methyl), 0.97 (methylene) and N—H = 0.86 Å, U_{iso}(H) = 1.2 or 1.5 U_{eq}(C, N).

Figures

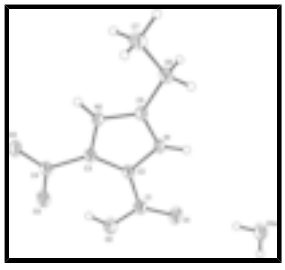


Fig. 1. The structure of the title compound, showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids.

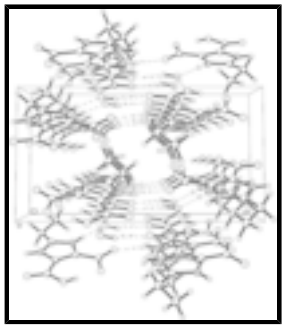


Fig. 2. A view of the three-dimensional network constructed by O—H···O and N—H···O hydrogen bonding interactions

2-Ethyl-1*H*-imidazole-4-carboxylate monohydrate

Crystal data

$C_7H_{10}N_2O_5$	$F(000) = 424$
$M_r = 202.17$	$D_x = 1.560$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1702 reflections
$a = 7.6132$ (6) Å	$\theta = 2.5$–$25.9°$
$b = 14.3779$ (16) Å	$\mu = 0.13$ mm^{-1}
$c = 7.9396$ (8) Å	$T = 298$ K
$\beta = 97.799$ (1)°	Block, colorless
$V = 861.04$ (15) Å^3	0.50 × 0.41 × 0.40 mm
$Z = 4$	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	1510 independent reflections
Radiation source: fine-focus sealed tube	1166 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.031$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)	$h = -9 \rightarrow 8$
$T_{min} = 0.936$, $T_{max} = 0.948$	$k = -17 \rightarrow 13$
4224 measured reflections	$l = -9 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1510 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
129 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
3 restraints	Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.060 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.4705 (2)	0.42162 (11)	0.2301 (2)	0.0314 (4)
H1	0.4860	0.4805	0.2208	0.038*
N2	0.3607 (2)	0.29056 (11)	0.2927 (2)	0.0305 (4)
H2	0.2932	0.2499	0.3308	0.037*
O1	0.7740 (2)	0.46097 (10)	0.0811 (2)	0.0517 (5)
O2	0.8238 (2)	0.31103 (10)	0.0434 (2)	0.0457 (5)
H2A	0.7770	0.2619	0.0653	0.069*
O3	0.6974 (2)	0.16119 (9)	0.1124 (2)	0.0433 (4)
O4	0.4800 (2)	0.11124 (9)	0.2502 (2)	0.0436 (4)
O1W	0.8455 (2)	0.65550 (10)	0.0862 (2)	0.0501 (5)
H1W	0.9476	0.6582	0.0543	0.075*
H2W	0.8189	0.5995	0.1057	0.075*
C1	0.7364 (3)	0.37964 (14)	0.0950 (3)	0.0356 (5)
C2	0.5789 (3)	0.35434 (13)	0.1777 (2)	0.0295 (5)
C3	0.5090 (3)	0.27125 (13)	0.2173 (2)	0.0282 (5)
C4	0.5648 (3)	0.17372 (13)	0.1935 (3)	0.0314 (5)

C5	0.3383 (3)	0.38238 (13)	0.2972 (3)	0.0310 (5)
C6	0.1908 (3)	0.43371 (15)	0.3579 (3)	0.0466 (6)
H6A	0.1212	0.4632	0.2610	0.056*
H6B	0.2403	0.4827	0.4340	0.056*
C7	0.0694 (3)	0.37559 (17)	0.4485 (3)	0.0528 (7)
H7A	0.0221	0.3257	0.3758	0.079*
H7B	−0.0259	0.4135	0.4773	0.079*
H7C	0.1345	0.3504	0.5503	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0332 (9)	0.0177 (8)	0.0455 (11)	0.0005 (7)	0.0140 (8)	0.0015 (7)
N2	0.0316 (9)	0.0200 (8)	0.0425 (10)	−0.0005 (6)	0.0145 (8)	0.0011 (7)
O1	0.0503 (10)	0.0288 (9)	0.0821 (13)	−0.0062 (7)	0.0310 (9)	0.0027 (8)
O2	0.0437 (9)	0.0295 (8)	0.0707 (11)	0.0011 (7)	0.0321 (8)	0.0009 (7)
O3	0.0479 (9)	0.0275 (8)	0.0601 (10)	0.0068 (6)	0.0272 (8)	−0.0009 (7)
O4	0.0460 (9)	0.0202 (7)	0.0690 (11)	0.0006 (6)	0.0236 (8)	0.0029 (7)
O1W	0.0479 (10)	0.0283 (8)	0.0813 (12)	−0.0006 (7)	0.0344 (9)	−0.0023 (8)
C1	0.0344 (11)	0.0281 (11)	0.0466 (13)	0.0005 (9)	0.0137 (10)	0.0017 (9)
C2	0.0306 (11)	0.0234 (10)	0.0355 (11)	0.0029 (8)	0.0087 (9)	−0.0001 (8)
C3	0.0296 (10)	0.0222 (10)	0.0340 (11)	0.0015 (8)	0.0090 (9)	−0.0005 (8)
C4	0.0337 (11)	0.0227 (10)	0.0389 (12)	0.0023 (8)	0.0092 (9)	−0.0002 (9)
C5	0.0313 (11)	0.0222 (10)	0.0414 (12)	0.0011 (8)	0.0124 (9)	0.0005 (9)
C6	0.0433 (13)	0.0273 (12)	0.0751 (17)	0.0048 (9)	0.0295 (13)	−0.0017 (11)
C7	0.0468 (14)	0.0451 (14)	0.0734 (17)	0.0080 (11)	0.0334 (13)	0.0037 (13)

Geometric parameters (Å, °)

N1—C5	1.327 (2)	O1W—H2W	0.8501
N1—C2	1.372 (2)	C1—C2	1.488 (3)
N1—H1	0.8600	C2—C3	1.362 (3)
N2—C5	1.332 (3)	C3—C4	1.485 (3)
N2—C3	1.376 (2)	C5—C6	1.478 (3)
N2—H2	0.8600	C6—C7	1.500 (3)
O1—C1	1.212 (2)	C6—H6A	0.9700
O2—C1	1.288 (2)	C6—H6B	0.9700
O2—H2A	0.8200	C7—H7A	0.9600
O3—C4	1.281 (2)	C7—H7B	0.9600
O4—C4	1.227 (2)	C7—H7C	0.9600
O1W—H1W	0.8500		
C5—N1—C2	110.02 (16)	O4—C4—C3	118.07 (17)
C5—N1—H1	125.0	O3—C4—C3	117.11 (17)
C2—N1—H1	125.0	N1—C5—N2	107.67 (16)
C5—N2—C3	109.11 (15)	N1—C5—C6	124.74 (17)
C5—N2—H2	125.4	N2—C5—C6	127.55 (17)
C3—N2—H2	125.4	C5—C6—C7	115.01 (18)
C1—O2—H2A	109.5	C5—C6—H6A	108.5

H1W—O1W—H2W	110.3	C7—C6—H6A	108.5
O1—C1—O2	124.86 (19)	C5—C6—H6B	108.5
O1—C1—C2	119.35 (18)	C7—C6—H6B	108.5
O2—C1—C2	115.80 (17)	H6A—C6—H6B	107.5
C3—C2—N1	106.15 (16)	C6—C7—H7A	109.5
C3—C2—C1	132.84 (18)	C6—C7—H7B	109.5
N1—C2—C1	121.01 (17)	H7A—C7—H7B	109.5
C2—C3—N2	107.05 (16)	C6—C7—H7C	109.5
C2—C3—C4	132.14 (17)	H7A—C7—H7C	109.5
N2—C3—C4	120.81 (17)	H7B—C7—H7C	109.5
O4—C4—O3	124.82 (17)		
C5—N1—C2—C3	−0.7 (2)	C5—N2—C3—C4	−179.30 (18)
C5—N1—C2—C1	179.02 (18)	C2—C3—C4—O4	176.4 (2)
O1—C1—C2—C3	−176.8 (2)	N2—C3—C4—O4	−3.4 (3)
O2—C1—C2—C3	2.9 (4)	C2—C3—C4—O3	−4.6 (3)
O1—C1—C2—N1	3.5 (3)	N2—C3—C4—O3	175.56 (18)
O2—C1—C2—N1	−176.74 (19)	C2—N1—C5—N2	1.3 (2)
N1—C2—C3—N2	−0.1 (2)	C2—N1—C5—C6	−176.3 (2)
C1—C2—C3—N2	−179.8 (2)	C3—N2—C5—N1	−1.3 (2)
N1—C2—C3—C4	−179.9 (2)	C3—N2—C5—C6	176.2 (2)
C1—C2—C3—C4	0.4 (4)	N1—C5—C6—C7	−172.7 (2)
C5—N2—C3—C2	0.9 (2)	N2—C5—C6—C7	10.2 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	0.86	1.91	2.754 (2)	168
N2—H2···O1W^{ii}	0.86	1.89	2.751 (2)	177
O2—H2A···O3	0.82	1.63	2.452 (2)	176
O1W—H1W···O2^{iii}	0.85	2.05	2.8863 (19)	169
O1W—H2W···O1	0.85	2.03	2.849 (2)	163

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*+2, −*y*+1, −*z*.

Fig. 1

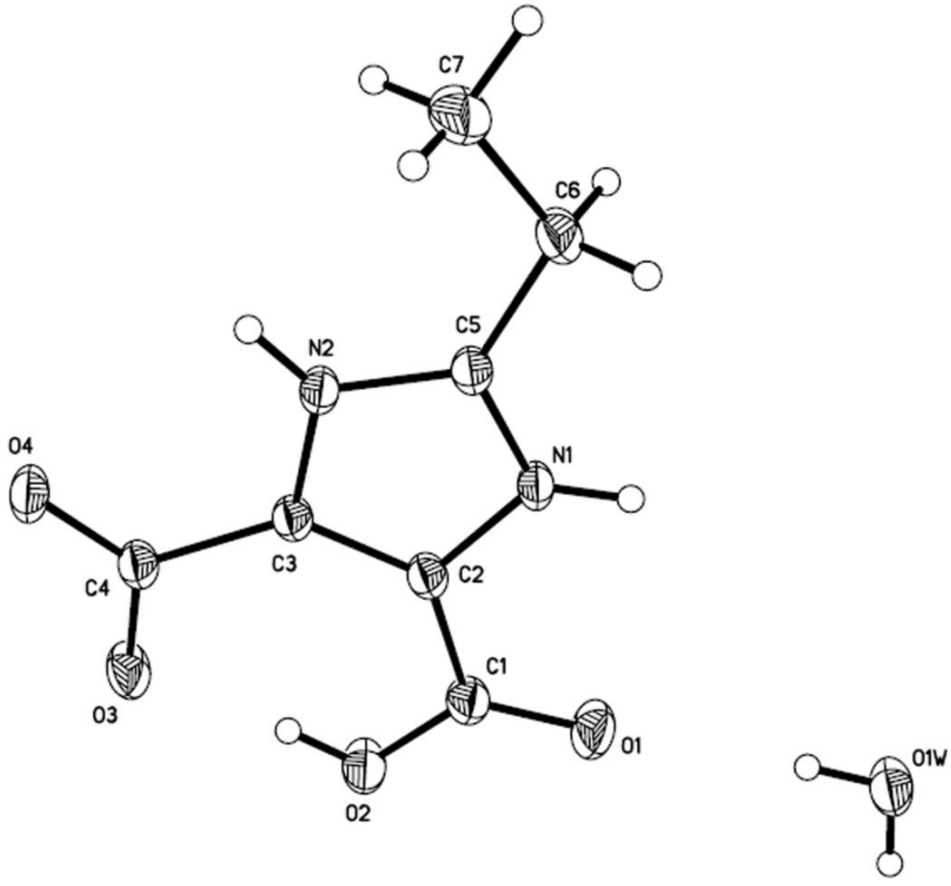


Fig. 2