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5-Carboxy-2-isopropyl-1*H*-imidazol-3-ium-4-carboxylate monohydrate

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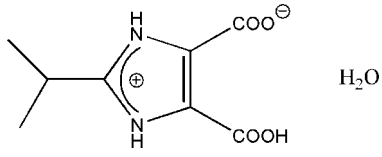
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.123; data-to-parameter ratio = 15.3.

In the title compound, $C_8H_{10}N_2O_4\cdot H_2O$, the imidazole N atom is protonated and one of the carboxylate groups is deprotoned, forming a zwitterion. An intramolecular O—H···O hydrogen bond occurs. The crystal structure is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds. In addition, intermolecular N—H···O and O—H···O hydrogen bonds link the molecules into two-dimensional networks parallel to $(10\bar{2})$.

Related literature

For the use of related imidazoledicarboxylic acid structures in coordination chemistry, see: Sun *et al.* (2006); Merchan & Stoeckli-Evans (2007); Guo (2009); Wang & Qin (2010); Wang *et al.* (2010); Feng *et al.* (2010); Li *et al.* (2010). For the synthesis of the title compound, see: Alcalde *et al.* (1992).



H_2O

Experimental

Crystal data

$C_8H_{10}N_2O_4\cdot H_2O$
M_r = 216.20
Monoclinic, $P2_1/c$
a = 7.828 (2) Å
b = 14.308 (4) Å
c = 8.930 (2) Å
β = 93.590 (3)°
V = 998.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.40 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.953, T_{max} = 0.967
4874 measured reflections
2147 independent reflections
1599 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.06
2147 reflections
140 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.82	1.64	2.4576 (19)	175
N1—H1···O5	0.86	1.83	2.6879 (19)	171
N2—H2···O1^{i}	0.86	1.93	2.7619 (19)	162
O5—H5*B*···O3^{ii}	0.85	2.03	2.8684 (19)	171
O5—H5*A*···O4^{iii}	0.85	1.94	2.7857 (19)	173

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2015).

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supplementary materials

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5-Carboxy-2-isopropyl-1*H*-imidazol-3-ium-4-carboxylate monohydrate

C.-J. Du, Z.-H. Shi, L.-S. Wang and C.-L. Du

Comment

In the past the construction of metal complexes based on N-heterocyclic carboxylic acids has attracted much attention due to their intriguing topologies as well as their potential applications in many fields. Particular attention has been paid to the 1*H*-imidazole-4,5-dicarboxylic acid ligand and its analogs: (Sun *et al.*, 2006) have synthesized the 4-carboxy-2-(pyridinium-4-yl)-1*H*-imidazole-5-carboxylate monohydrate; Merchan *et al.* (2007) have prepared the dimethylammonium 4-carboxy-1*H*-imidazole-5-carboxylate; (Guo, 2009) have reported the 4-carboxy-2-methyl-1*H*-imidazole-5-carboxylate monohydrate and (Wang & Qin, 2010) have reported the dimethylammonium 4-carboxy-2-n-propyl-1*H*-imidazole-5-carboxylate. All of these 1*H*-imidazole-4,5-dicarboxylic acid and their analogs have been used as ligands to design metal complexes and most of them are proved ideal ligands (Wang *et al.*, 2010; Feng *et al.*, 2010; Li *et al.*, 2010). However, the crystal structure of 4-carboxy-2-isopropyl-1*H*-imidazole-5-carboxylate has not been yet determined. Keeping that in mind, we report here the preparation and crystal structure of the title compound. The crystal structure (Fig.2, Table1) is stabilized by two intramolecular and three intermolecular N—H···O and O—H···O hydrogen bonds which link the molecules into two-dimensional networks parallel to the $(10\bar{2})$ planes.

Experimental

The title compound was synthesized according to the method reported in the literature (Alcalde *et al.*, 1992). Colourless single crystals suitable for X-ray diffraction were obtained by slow evaporation of a water solution of the compound.

Refinement

H atoms bonded to the water O atom were located in an electron density map and refined with distance restraints of O—H = 0.85 Å. Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.96—0.98 Å, N—H = 0.86 Å and O—H = 0.82 Å. U_{iso}(H) = kU_{eq}(carrier atom), where k = 1.2 for N and $C_{tertiary}$ and 1.5 for O and C_{methyl}.

Figures

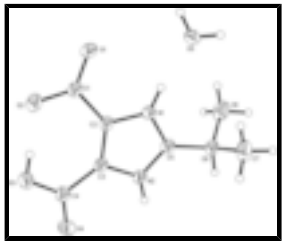

Fig. 1. The title compound with displacement ellipsoids drawn at the 30% probability level.

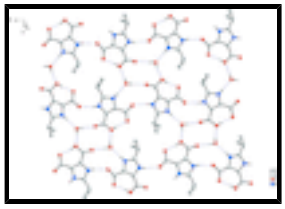

Fig. 2. A view of the crystal structure. The H-atoms not included in hydrogen bonding have been omitted for clarity.

5-Carboxy-2-isopropyl-1*H*-imidazol-3-ium-4-carboxylate monohydrate

Crystal data

$C_8H_{10}N_2O_4·H_2O$	$F(000) = 456$
$M_r = 216.20$	$D_x = 1.439$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1379 reflections
$a = 7.828$ (2) Å	$\theta = 2.7–25.1°$
$b = 14.308$ (4) Å	$\mu = 0.12$ mm^{-1}
$c = 8.930$ (2) Å	$T = 298$ K
$\beta = 93.590$ (3)°	Block, colourless
$V = 998.2$ (4) Å^3	0.40 × 0.32 × 0.28 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2147 independent reflections
Radiation source: fine-focus sealed tube	1599 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.028$
φ and ω scans	$\theta_{max} = 27.0°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -9\rightarrow9$
$T_{min} = 0.953$, $T_{max} = 0.967$	$k = -17\rightarrow18$
4874 measured reflections	$l = -11\rightarrow7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2147 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
140 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
3 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.016 (3)

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$F^2 > 2\mathrm{sigma}(F^2)$ 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}
C4	1.2174 (2)	0.88114 (12)	0.4485 (2)	0.0397 (4)
C2	1.0189 (2)	0.76673 (11)	0.29253 (18)	0.0346 (4)
C3	1.0710 (2)	0.85223 (11)	0.34578 (19)	0.0350 (4)
C6	0.6833 (2)	0.91545 (12)	0.1177 (2)	0.0413 (4)
H6	0.6971	0.9835	0.1205	0.050*
C1	1.0889 (2)	0.67085 (11)	0.3136 (2)	0.0391 (4)
C5	0.8360 (2)	0.87217 (11)	0.19793 (19)	0.0366 (4)
C7	0.5240 (2)	0.89011 (17)	0.1978 (2)	0.0593 (6)
H7A	0.5139	0.8233	0.2031	0.089*
H7B	0.4249	0.9154	0.1435	0.089*
H7C	0.5328	0.9156	0.2975	0.089*
C8	0.6644 (3)	0.88445 (16)	−0.0461 (2)	0.0622 (6)
H8A	0.7620	0.9051	−0.0971	0.093*
H8B	0.5624	0.9113	−0.0934	0.093*
H8C	0.6571	0.8175	−0.0507	0.093*
O2	1.21461 (18)	0.66102 (9)	0.41079 (17)	0.0588 (4)
O4	1.24232 (16)	0.96417 (9)	0.47291 (15)	0.0527 (4)
O3	1.31065 (17)	0.81470 (9)	0.50578 (16)	0.0521 (4)
H3	1.2783	0.7647	0.4692	0.078*
O1	1.02199 (16)	0.60794 (8)	0.23815 (16)	0.0494 (4)
N1	0.87377 (17)	0.78132 (9)	0.20091 (15)	0.0368 (3)
H1	0.8162	0.7384	0.1529	0.044*
N2	0.95551 (16)	0.91583 (9)	0.28360 (16)	0.0372 (4)
H2	0.9601	0.9752	0.2981	0.045*
O5	0.66848 (18)	0.65027 (9)	0.07026 (18)	0.0638 (5)
H5B	0.5604	0.6549	0.0562	0.096*
H5A	0.6938	0.5927	0.0646	0.096*

Atomic displacement parameters (Å^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

C4	0.0340 (9)	0.0334 (10)	0.0514 (10)	0.0000 (7)	0.0003 (7)	−0.0017 (8)
C2	0.0306 (8)	0.0264 (8)	0.0464 (9)	0.0013 (6)	−0.0014 (7)	0.0008 (7)
C3	0.0308 (8)	0.0258 (8)	0.0483 (10)	0.0020 (6)	0.0007 (7)	0.0004 (6)
C6	0.0366 (9)	0.0297 (9)	0.0565 (11)	0.0066 (7)	−0.0047 (7)	0.0023 (7)
C1	0.0346 (9)	0.0256 (9)	0.0566 (10)	0.0024 (7)	−0.0013 (7)	0.0012 (7)
C5	0.0336 (9)	0.0260 (8)	0.0499 (10)	0.0017 (7)	−0.0005 (7)	0.0001 (7)
C7	0.0405 (11)	0.0786 (16)	0.0581 (12)	0.0123 (10)	−0.0015 (9)	0.0096 (11)
C8	0.0637 (13)	0.0724 (16)	0.0502 (12)	0.0243 (12)	0.0007 (10)	0.0104 (10)
O2	0.0564 (9)	0.0348 (8)	0.0813 (10)	0.0108 (6)	−0.0258 (7)	0.0023 (6)
O4	0.0521 (8)	0.0331 (7)	0.0711 (9)	−0.0084 (6)	−0.0102 (6)	−0.0041 (6)
O3	0.0438 (8)	0.0372 (7)	0.0725 (9)	0.0015 (6)	−0.0178 (6)	−0.0031 (6)
O1	0.0450 (7)	0.0245 (6)	0.0775 (9)	0.0024 (5)	−0.0040 (6)	−0.0058 (6)
N1	0.0336 (7)	0.0242 (7)	0.0514 (8)	0.0017 (6)	−0.0070 (6)	−0.0022 (6)
N2	0.0334 (8)	0.0205 (7)	0.0570 (9)	0.0013 (5)	−0.0028 (6)	−0.0026 (6)
O5	0.0516 (8)	0.0357 (8)	0.0994 (11)	0.0046 (6)	−0.0323 (8)	−0.0154 (7)

Geometric parameters (Å, °)

C4—O4	1.221 (2)	C5—N2	1.327 (2)
C4—O3	1.285 (2)	C5—N1	1.333 (2)
C4—C3	1.481 (2)	C7—H7A	0.9600
C2—C3	1.366 (2)	C7—H7B	0.9600
C2—N1	1.374 (2)	C7—H7C	0.9600
C2—C1	1.485 (2)	C8—H8A	0.9600
C3—N2	1.375 (2)	C8—H8B	0.9600
C6—C5	1.489 (2)	C8—H8C	0.9600
C6—C7	1.520 (3)	O3—H3	0.8200
C6—C8	1.526 (3)	N1—H1	0.8600
C6—H6	0.9800	N2—H2	0.8600
C1—O1	1.222 (2)	O5—H5B	0.8500
C1—O2	1.279 (2)	O5—H5A	0.8501
O4—C4—O3	124.60 (17)	C6—C7—H7A	109.5
O4—C4—C3	119.41 (16)	C6—C7—H7B	109.5
O3—C4—C3	115.99 (15)	H7A—C7—H7B	109.5
C3—C2—N1	106.79 (14)	C6—C7—H7C	109.5
C3—C2—C1	133.16 (16)	H7A—C7—H7C	109.5
N1—C2—C1	120.04 (15)	H7B—C7—H7C	109.5
C2—C3—N2	106.13 (14)	C6—C8—H8A	109.5
C2—C3—C4	131.93 (15)	C6—C8—H8B	109.5
N2—C3—C4	121.93 (14)	H8A—C8—H8B	109.5
C5—C6—C7	109.35 (15)	C6—C8—H8C	109.5
C5—C6—C8	111.52 (14)	H8A—C8—H8C	109.5
C7—C6—C8	110.41 (17)	H8B—C8—H8C	109.5
C5—C6—H6	108.5	C4—O3—H3	109.5
C7—C6—H6	108.5	C5—N1—C2	109.54 (14)
C8—C6—H6	108.5	C5—N1—H1	125.2
O1—C1—O2	125.32 (16)	C2—N1—H1	125.2
O1—C1—C2	117.95 (16)	C5—N2—C3	110.08 (14)
O2—C1—C2	116.73 (15)	C5—N2—H2	125.0

N2—C5—N1	107.44 (14)	C3—N2—H2	125.0
N2—C5—C6	126.67 (15)	H5B—O5—H5A	107.5
N1—C5—C6	125.84 (15)		
N1—C2—C3—N2	−0.31 (17)	C7—C6—C5—N2	−106.3 (2)
C1—C2—C3—N2	178.46 (17)	C8—C6—C5—N2	131.29 (19)
N1—C2—C3—C4	179.79 (16)	C7—C6—C5—N1	71.0 (2)
C1—C2—C3—C4	−1.4 (3)	C8—C6—C5—N1	−51.3 (2)
O4—C4—C3—C2	175.88 (18)	N2—C5—N1—C2	1.04 (18)
O3—C4—C3—C2	−3.9 (3)	C6—C5—N1—C2	−176.75 (15)
O4—C4—C3—N2	−4.0 (2)	C3—C2—N1—C5	−0.44 (18)
O3—C4—C3—N2	176.25 (16)	C1—C2—N1—C5	−179.40 (15)
C3—C2—C1—O1	−172.81 (18)	N1—C5—N2—C3	−1.24 (18)
N1—C2—C1—O1	5.8 (2)	C6—C5—N2—C3	176.52 (16)
C3—C2—C1—O2	7.7 (3)	C2—C3—N2—C5	0.97 (18)
N1—C2—C1—O2	−173.71 (15)	C4—C3—N2—C5	−179.12 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.82	1.64	2.4576 (19)	175
N1—H1···O5	0.86	1.83	2.6879 (19)	171
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Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+1/2; (ii) *x*−1, −*y*+3/2, *z*−1/2; (iii) −*x*+2, *y*−1/2, −*z*+1/2.

Fig. 1

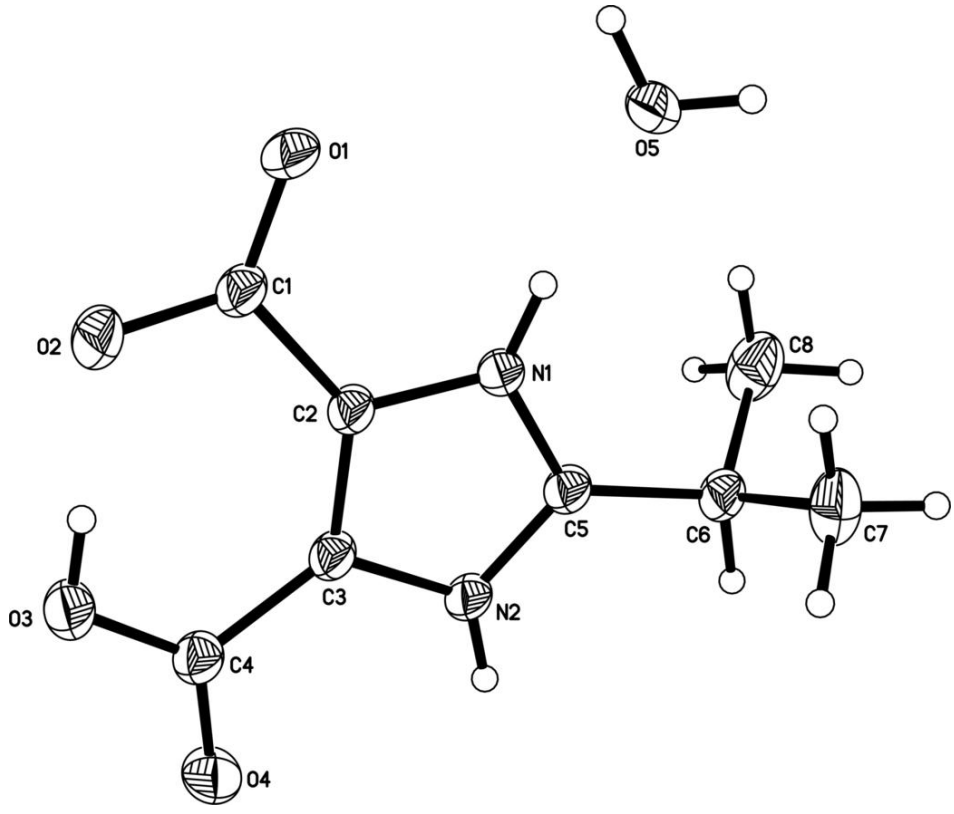

Fig. 2