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1-Methyl-4-nitro-1*H*-imidazole-2-carboxylic acid dihydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.109
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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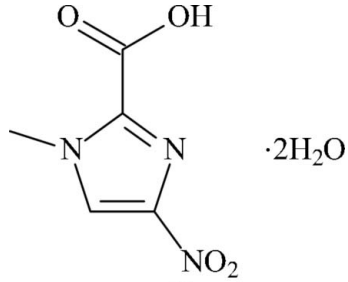
The hydrolysis of ethyl 1-methyl-4-nitro-1*H*-imidazole-2-carboxylate leads to 1-methyl-4-nitro-1*H*-imidazol-2-carboxylic acid, which crystallizes as a dihydrate, $C_5H_5N_3O_4{\cdot}2H_2O$. The planar organic entity is a flat molecule; the molecules are linked through the water molecules by hydrogen bonding into a three-dimensional network.

Comment

Ethyl 1-methyl-4-nitro-1*H*-imidazole-2-carboxylate, a reagent that is used for the synthesis of the analogs of netropsin and distamycin, exists as a molecule that is disordered in the carboxyethyl substituent. The disorder is the consequence of packing (Wu *et al.*, 2004). Hydrolysis of this ester yields the title carboxylic acid, which crystallizes as a dihydrate, (I) (Fig. 1). The acid molecule is planar; the molecules interact with the water molecules to form a three-dimensional hydrogen-bonded network (Fig. 2 and Table 2).



(I)

Experimental

A small quantity of the ethyl ester of the title carboxylic acid (Wu *et al.*, 2004) was suspended in lithium hydroxide solution (0.1 *M*) and tetrahydrofuran (THF) was added until it dissolved; the water/THF ratio was approximately 1:1. The mixture was stirred for 30 min. The solvent was removed and the product recrystallized from diethyl ether.

Crystal data

$C_5H_5N_3O_4{\cdot}2H_2O$
M_r = 207.15
Monoclinic, $P2_1/c$
a = 7.5163 (9) Å
b = 12.199 (2) Å
c = 9.730 (1) Å
β = 97.735 (2)°
V = 884.1 (2) Å^3
Z = 4
D_x = 1.556 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 890 reflections
θ = 2.7–27.0°
μ = 0.14 mm^{-1}
T = 295 (2) K
Yellow, prism
0.50 × 0.24 × 0.19 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
5110 measured reflections
1931 independent reflections

1504 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.0^{\circ}$
$h = -9 \rightarrow 8$
$k = -15 \rightarrow 14$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.109$
$S = 1.02$
1931 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.1428P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.299 (2)	N2—C4	1.348 (2)
O2—C1	1.203 (2)	N2—C2	1.365 (2)
O4—N3	1.220 (2)	N2—C3	1.470 (2)
O3—N3	1.217 (2)	N3—C5	1.431 (2)
N1—C2	1.321 (2)	C1—C2	1.485 (2)
N1—C5	1.352 (2)	C4—C5	1.365 (2)
C2—N1—C5	103.7 (1)	O1—C1—C2	112.1 (1)
C4—N2—C2	107.2 (1)	N1—C2—N2	111.9 (1)
C4—N2—C3	124.0 (1)	N1—C2—C1	123.9 (1)
C2—N2—C3	128.8 (1)	N2—C2—C1	124.2 (1)
O3—N3—O4	123.8 (1)	N2—C4—C5	104.9 (1)
O3—N3—C5	117.6 (1)	N1—C5—C4	112.3 (1)
O4—N3—C5	118.6 (1)	N1—C5—N3	121.7 (1)
O2—C1—O1	125.6 (1)	C4—C5—N3	126.0 (1)
O2—C1—C2	122.4 (1)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2w^{i}	0.82	1.73	2.546 (2)	176
O1w—H1w1···N1	0.85 (1)	2.02 (1)	2.868 (2)	179 (2)
O1w—H1w2···O4^{i}	0.84 (1)	2.33 (1)	3.134 (2)	161 (2)
O2w—H2w1···O1w	0.85 (1)	1.93 (1)	2.754 (2)	165 (2)
O2w—H2w2···O1w^{ii}	0.84 (1)	2.01 (1)	2.848 (2)	173 (2)
C4—H4···O2^{iii}	0.93	2.32	3.206 (2)	158

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

The aromatic, methyl and acid H atoms were placed at calculated positions in the riding-model approximation (C—$H_{aromatic}$ = 0.93 Å, C—H_{methyl} = 0.96 Å and O—H = 0.82 Å); the methyl and OH groups were rotated to fit the electron density. The water H atoms were located in a difference Fourier map, and were refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å. The displacement parameters of all H atoms were refined freely.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

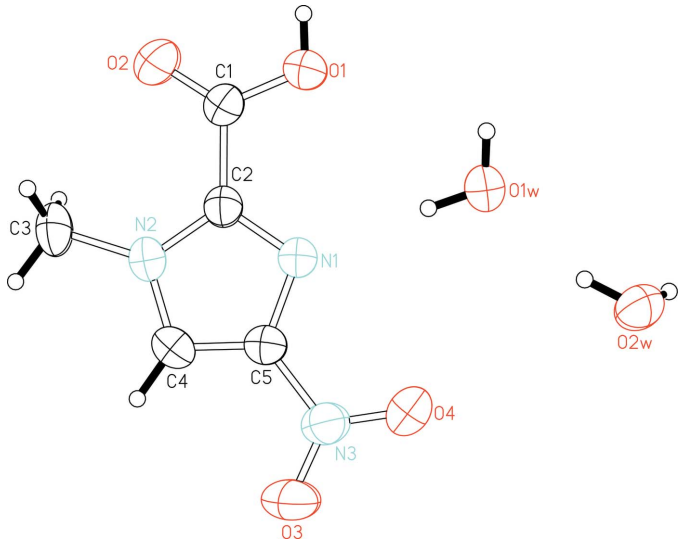


Figure 1
ORTEPII (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

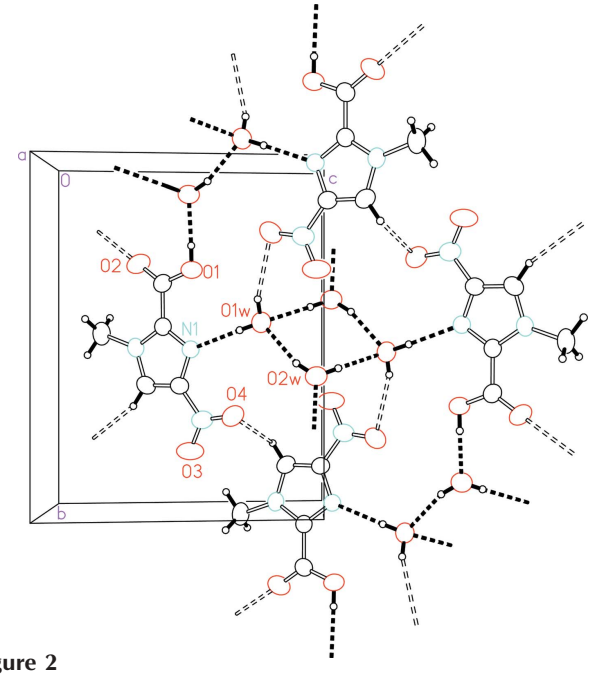


Figure 2
Detail of the strong (solid dashed lines) and weak (hollow dashed lines) hydrogen-bonding interactions in (I).

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