organic compounds

2-(1H-Tetrazol-1-yl)acetic acid monohydrate

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The crystal structure of the title compound, $C_3H_4N_4O_2 \cdot H_2O$, exhibits O—H···O and O—H···N hydrogen bonds, which lead to the formation of a two-dimensional network parallel to the bc plane. The dihedral angle between the ring and the carboxylic acid group is 84.6 (14)°.

Related literature

For the use of 2-(1H-tetrazol-1-yl) acetic acid as a pharmaceutical intermediate, see: Gunnlaugsson & Stomeo (2007). For its coordination properties, see: Ghosh & Bharadwaj (2004). For the synthesis, see: Jústiz et al. (1997).
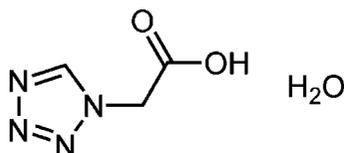
Experimental

Crystal data

$C_3H_4N_4O_2 \cdot H_2O$	b = 5.1871 (10) Å
M_r = 146.12	c = 9.874 (2) Å
Orthorhombic, $Pna2_1$	V = 646.2 (2) Å^3
a = 12.618 (3) Å	Z = 4

Mo $K\alpha$ radiation	T = 293 K
μ = 0.13 mm^{-1}	$0.26 \times 0.23 \times 0.19$ mm

Data collection

Rigaku SCXmini diffractometer	6216 measured reflections
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	786 independent reflections
T_{min} = 0.965, T_{max} = 0.983	715 reflections with $I > 2\sigma(I)$
	R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.069	
S = 1.17	
786 reflections	$\Delta\rho_{max}$ = 0.11 e Å^{-3}
103 parameters	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···N2	0.83 (4)	2.11 (4)	2.864 (3)	150 (3)
O1—H1A···O3^i	0.89 (4)	1.72 (4)	2.603 (3)	175 (4)
O3—H3A···O2^{ii}	0.76 (3)	2.00 (3)	2.752 (3)	168 (3)

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

Data collection: CrystalClear (Rigaku, 2005); cell refinement: CrystalClear; data reduction: CrystalClear; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: SHELXL97.

References

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Gunnlaugsson, T. & Stomeo, F. (2007). Org. Biomol. Chem. **5**, 1999–2009.
Jústiz, O. H., Fernández-Lafuente, R. & Guisán, J. M. (1997). J. Org. Chem. **62**, 9099–9106.
Rigaku (2005). CrystalClear. Rigaku Corporation, Tokyo, Japan.
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supporting information

2-(1*H*-Tetrazol-1-yl)acetic acid monohydrate

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S1. Comment

2-(1*H*-tetrazol-1-yl) acetic acid is an important raw material to prepare a variety of antibiotic drugs, and is widely used as an important pharmaceutical intermediate (Gunnlaugsson & Stomeo, 2007). In recent years, researchers found that tetrazole acetic acid has excellent coordination properties (Ghosh & Bharadwaj, 2004). We report here the crystal structure of the title compound (Fig. 1).

In the title compound the carboxyl functional group is almost perpendicular to the tetrazole heterocycle with a dihedral angle of 87.3 (2)°. The O3—H3B···N2 hydrogen bond anchors the water molecule to the tetrazole heterocycle. Two intermolecular hydrogen bonds (O1—H1A···O3 and O3—H3A···O2) connect the water molecule and two carboxyl groups from the neighboring asymmetric unit, forming layers with louver-like network (Fig. 2).

S2. Experimental

A procedure similar to the previously published method of Jústiz *et al.* (1997) was applied. To a solution of 2-aminoacetic acid (7.5 g, 0.1 mol) in 50 ml acetic acid was added triethoxymethane (32.4 g, 0.22 mol) and sodium azide (7.15 g, 0.11 mol). The mixture was refluxed for 3.0 h at 80°C. Active carbon was used to discolor the mixture, which was then refluxed for another 10 minutes. Heating was stopped and cooled to room temperature, the mixture was filtered to remove the active carbon and concentrated hydrochloric acid was trickled into the filtrate and a white solid product precipitated out. The precipitate was extracted by ethyl acetate and washed with saturated solution of sodium bicarbonate, brine, and then dried over MgSO$_4$. Evaporation of the solvent in vacuum afforded the 2-(1*H*-tetrazol-1-yl) acetic acid compound. The pale yellow single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of a solution in the 3:7 (v/v) mixture of petroleum ether and ethyl acetate.

S3. Refinement

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. Hydrogen atom positions were calculated geometrically and were set to ride on the associated C atoms, with U_{iso}(H)= 1.2 U_{iso}(C). The H atoms on O were located in difference electron density maps and were refined freely with isotropic displacement parameters.
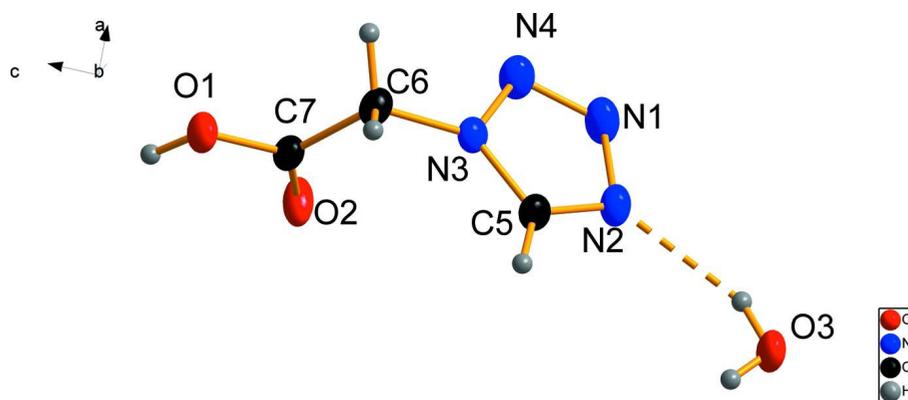
Figure 1

Asymmetric unit with anisotropic ellipsoid representation, shown with the atom labeling scheme. Ellispoids are shown at the 50% probability level.
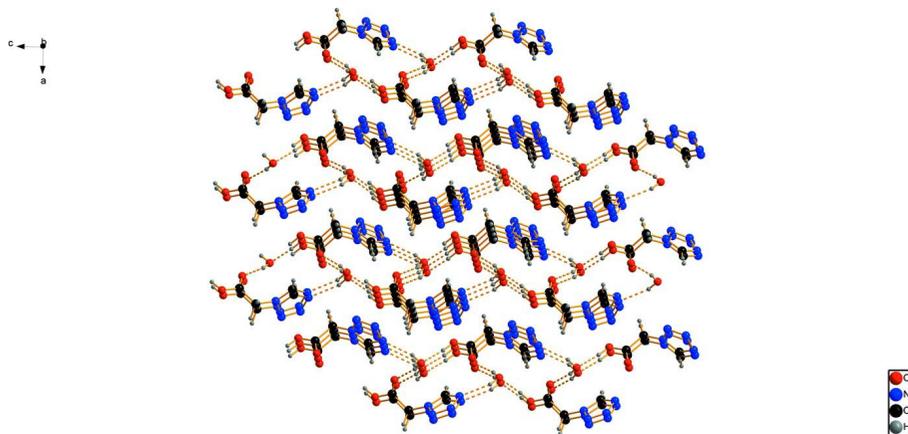
Figure 2

Packing diagram for the title compound. Hydrogen bonds are denoted by dashed lines.

2-(1*H*-Tetrazol-1-yl)acetic acid monohydrate

Crystal data

$C_3H_4N_4O_2 \cdot H_2O$	$F(000) = 304$
$M_r = 146.12$	$D_x = 1.502$ Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 6216 reflections
$a = 12.618$ (3) Å	$\theta = 3.2–27.5°$
$b = 5.1871$ (10) Å	$\mu = 0.13$ mm^{-1}
$c = 9.874$ (2) Å	$T = 293$ K
$V = 646.2$ (2) Å^3	Needle, pale yellow
$Z = 4$	$0.26 \times 0.23 \times 0.19$ mm

Data collection

Rigaku SCXmini diffractometer	Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
Radiation source: fine-focus sealed tube	$T_{min} = 0.965$, $T_{max} = 0.983$
Graphite monochromator	6216 measured reflections
ω scans	786 independent reflections

715 reflections with $I > 2\sigma(I)$	$h = -16 \rightarrow 16$
$R_{int} = 0.031$	$k = -6 \rightarrow 6$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.030$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.069$	
$S = 1.17$	H atoms treated by a mixture of independent and constrained refinement
786 reflections	
103 parameters	$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 0.0792P]$ where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.11$ e Å^{-3}
	$\Delta\rho_{min} = -0.12$ 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	0.38513 (14)	0.5364 (3)	1.01953 (17)	0.0450 (4)
O2	0.30421 (14)	0.8326 (3)	0.89450 (17)	0.0525 (5)
N2	0.37640 (16)	0.8301 (4)	0.4743 (2)	0.0436 (5)
C5	0.36860 (19)	0.6540 (4)	0.5678 (2)	0.0381 (5)
H5A	0.3281	0.5047	0.5614	0.046*
N4	0.47431 (18)	0.9471 (4)	0.6448 (2)	0.0497 (5)
C7	0.37025 (18)	0.6679 (4)	0.9087 (2)	0.0358 (5)
N1	0.44272 (18)	1.0105 (4)	0.5252 (2)	0.0527 (6)
N3	0.42748 (14)	0.7205 (3)	0.67377 (18)	0.0334 (4)
C6	0.44751 (19)	0.5888 (4)	0.8003 (2)	0.0379 (5)
H6A	0.5190	0.6270	0.8302	0.046*
H6B	0.4424	0.4042	0.7860	0.046*
O3	0.27445 (17)	0.7053 (4)	0.22354 (17)	0.0509 (5)
H1A	0.344 (3)	0.595 (6)	1.086 (4)	0.079 (11)*
H3A	0.246 (2)	0.600 (6)	0.263 (4)	0.062 (10)*
H3B	0.310 (3)	0.789 (6)	0.279 (4)	0.062 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0495 (9)	0.0525 (10)	0.0330 (9)	0.0046 (8)	−0.0002 (8)	0.0146 (8)

O2	0.0605 (10)	0.0645 (11)	0.0327 (9)	0.0259 (9)	0.0079 (8)	0.0131 (9)
N2	0.0554 (12)	0.0453 (11)	0.0303 (9)	−0.0005 (9)	0.0026 (9)	0.0036 (9)
C5	0.0438 (14)	0.0403 (12)	0.0303 (11)	−0.0033 (9)	0.0011 (9)	−0.0052 (9)
N4	0.0678 (14)	0.0421 (11)	0.0393 (11)	−0.0149 (10)	0.0003 (11)	−0.0023 (9)
C7	0.0409 (12)	0.0368 (11)	0.0298 (11)	−0.0010 (10)	−0.0039 (9)	0.0036 (10)
N1	0.0729 (14)	0.0454 (11)	0.0396 (12)	−0.0118 (10)	0.0050 (12)	0.0042 (10)
N3	0.0402 (9)	0.0325 (9)	0.0276 (8)	0.0000 (8)	0.0031 (8)	−0.0036 (7)
C6	0.0446 (12)	0.0366 (11)	0.0326 (11)	0.0070 (11)	−0.0046 (10)	−0.0011 (9)
O3	0.0612 (11)	0.0625 (12)	0.0289 (9)	−0.0116 (10)	−0.0018 (8)	0.0107 (9)

Geometric parameters (Å, º)

O1—C7	1.303 (3)	N4—N3	1.346 (3)
O1—H1A	0.89 (4)	C7—C6	1.505 (3)
O2—C7	1.202 (3)	N3—C6	1.446 (3)
N2—C5	1.303 (3)	C6—H6A	0.9700
N2—N1	1.352 (3)	C6—H6B	0.9700
C5—N3	1.329 (3)	O3—H3A	0.76 (3)
C5—H5A	0.9300	O3—H3B	0.83 (4)
N4—N1	1.289 (3)		
C7—O1—H1A	111 (2)	C5—N3—N4	107.74 (19)
C5—N2—N1	105.6 (2)	C5—N3—C6	130.92 (19)
N2—C5—N3	109.49 (19)	N4—N3—C6	121.31 (19)
N2—C5—H5A	125.3	N3—C6—C7	111.87 (17)
N3—C5—H5A	125.3	N3—C6—H6A	109.2
N1—N4—N3	106.37 (19)	C7—C6—H6A	109.2
O2—C7—O1	124.8 (2)	N3—C6—H6B	109.2
O2—C7—C6	124.0 (2)	C7—C6—H6B	109.2
O1—C7—C6	111.19 (19)	H6A—C6—H6B	107.9
N4—N1—N2	110.82 (19)	H3A—O3—H3B	107 (3)
N1—N2—C5—N3	0.1 (3)	N1—N4—N3—C6	178.50 (19)
N3—N4—N1—N2	−0.3 (3)	C5—N3—C6—C7	−92.1 (3)
C5—N2—N1—N4	0.1 (3)	N4—N3—C6—C7	90.3 (3)
N2—C5—N3—N4	−0.3 (3)	O2—C7—C6—N3	−4.4 (3)
N2—C5—N3—C6	−178.2 (2)	O1—C7—C6—N3	176.12 (18)
N1—N4—N3—C5	0.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···N2	0.83 (4)	2.11 (4)	2.864 (3)	150 (3)
O1—H1A···O3[i]	0.89 (4)	1.72 (4)	2.603 (3)	175 (4)
O3—H3A···O2[ii]	0.76 (3)	2.00 (3)	2.752 (3)	168 (3)

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