organic compounds

1-Benzyl-5-methyl-1H-1,2,3-triazole-4-carboxylic acid monohydrate

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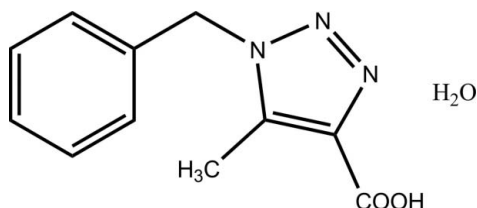Correspondence e-mail: zhaohong@seu.edu.cn

In the title compound, $C_{11}H_{11}N_3O_2 \cdot H_2O$, the planes of the triazole and phenyl rings are almost perpendicular to each other [dihedral angle 89.5 (3)°]. The crystal packing is stabilized by strong intermolecular O—H⋯O and O—H⋯N hydrogen bonds involving the water molecule as both donor and acceptor.

Related literature

For the synthesis of the title compound, see: El Khadem et al. (1968). For the biological activity of triazole compounds, see: Olesen et al. (2003); Tian et al. (2005). For related structures, see: Lin et al. (2008); Xiao et al. (2008).

Experimental

Crystal data

$C_{11}H_{11}N_3O_2 \cdot H_2O$	a = 6.5808 (13) Å
M_r = 235.24	b = 7.4995 (15) Å
Triclinic, $P\bar{1}$	c = 12.337 (3) Å
α = 99.87 (4)°	Mo $K\alpha$ radiation
β = 93.75 (3)°	μ = 0.10 mm^{-1}
γ = 91.80 (3)°	T = 292 K
V = 598.0 (2) Å^3	$0.35 \times 0.30 \times 0.25$ mm
Z = 2	

Data collection

Rigaku SCXmini diffractometer	6256 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	2730 independent reflections
T_{min} = 0.963, T_{max} = 0.976	1540 reflections with $I > 2\sigma(I)$
	R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064	156 parameters
$wR(F^2)$ = 0.174	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
2730 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1W—H1A⋯N1	0.92	1.96	2.870 (3)	172
O1W—H1B⋯O2^i	0.86	1.88	2.734 (3)	171
O1—H1⋯O1W^{ii}	0.82	1.75	2.563 (3)	168

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

El Khadem, H., Mansour, H. A. R. & Meshreki, M. H. (1968). *J. Chem. Soc. C*, pp. 1329–1331.
Lin, J. R., Yao, J. Y. & Zhao, H. (2008). *Acta Cryst.* E**64**, o1843.
Olesen, P. H., Sorensen, A. R., Urso, B., Kurtzhals, P., Bowler, A. N., Ehrbar, U. & Hansen, B. F. (2003). *J. Med. Chem.* **46**, 3333–3341.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tian, L., Sun, Y., Li, H., Zheng, X., Cheng, Y., Liu, X. & Qian, B. (2005). *J. Inorg. Biochem.* **99**, 1646–1652.
Xiao, J., Wang, W. X. & Zhao, H. (2008). *Acta Cryst.* E**64**, o2085.

supporting information

1-Benzyl-5-methyl-1*H*-1,2,3-triazole-4-carboxylic acid monohydrate

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S1. Comment

Triazole-related molecules have attracted considerable attention due to their biological activities (Olesen *et al.*, 2003; Tian *et al.*, 2005). Recently, we have reported the cerystal structure of a few triazole compounds (Lin *et al.* 2008; Xiao *et al.*2008). As an extension of our work on the structural characterization of the triazole-related compounds, we report herein the crystal structure of the title compound.
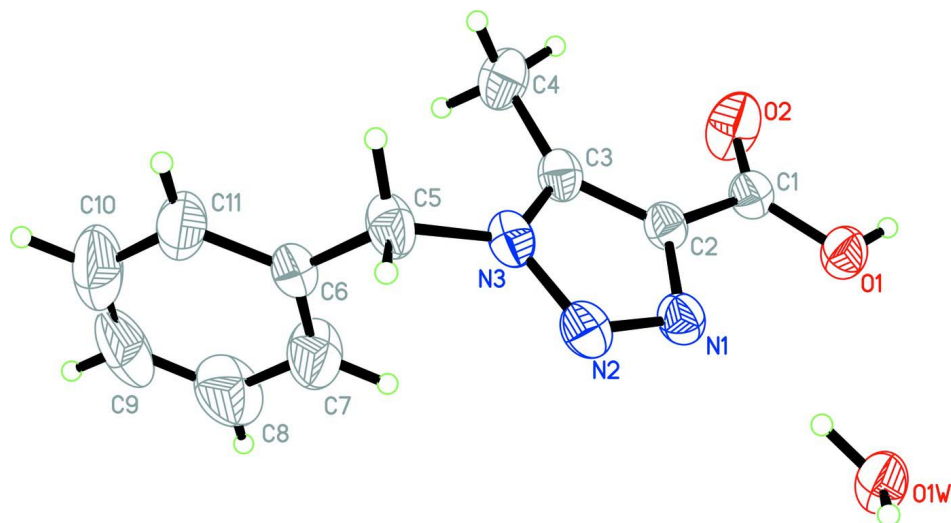
In the molecule of the title compound (Fig. 1), bond lengths and angles have normal values. The dihedral angle between the triazole and phenyl rings is 89.5 (3)°. The packing is stabilized by strong intermolecular O—H⋯O and O—H⋯N hydrogen bonds involving the triazole molecules and lattice water molecules (Fig.2; Table 1).

S2. Experimental

The title compound was prepared from azidomethylbenzene according to the reported method (El Khadem *et al.*, 1968). Colourless prismatic crystals suitable for X-ray analysis were obtained by slow evaporation of a 95% ethanol/water solution at room temperature.

S3. Refinement

The water and carboxylic H atoms were located from a difference Fourier map but not refined [U_{iso}(H) = 1.5U_{eq}(O)]. All other H atoms were fixed geometrically and treated as riding, with C—H = 0.93-0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms.

Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
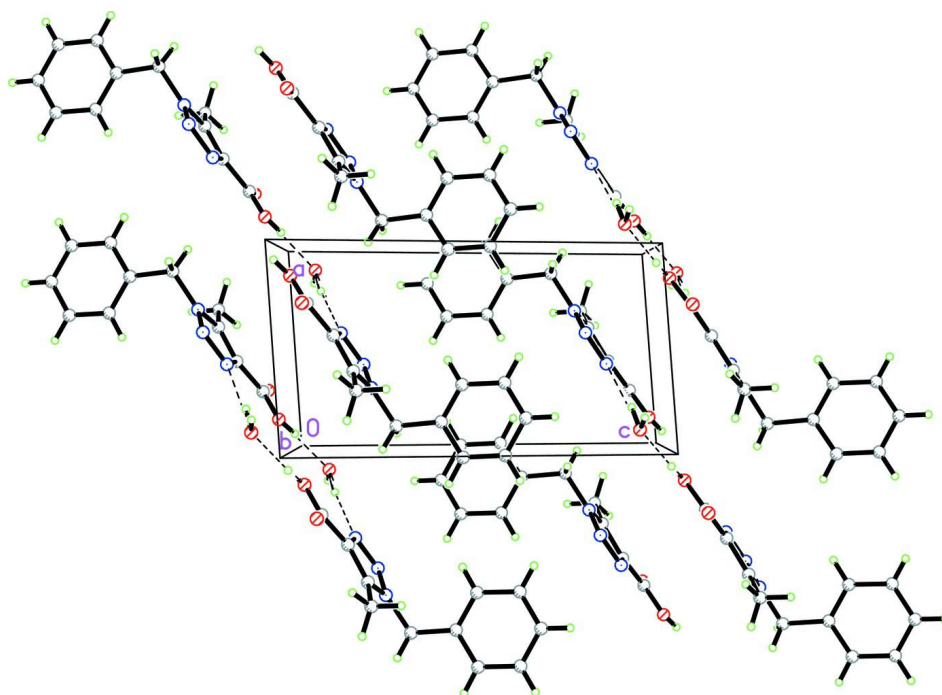
Figure 2

Packing diagram of the title compound viewed along the *b* axis. Intermolecular H bonds are shown as dashed lines.

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Crystal data

$C_{11}H_{11}N_3O_2 \cdot H_2O$	Hall symbol: -P 1
$M_r = 235.24$	$a = 6.5808\,(13)$ Å
Triclinic, $P\bar{1}$	$b = 7.4995\,(15)$ Å

$c = 12.337$ (3) Å
$\alpha = 99.87$ (4)°
$\beta = 93.75$ (3)°
$\gamma = 91.80$ (3)°
$V = 598.0$ (2) Å^3
$Z = 2$
$F(000) = 248$
$D_x = 1.306$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1986 reflections
$\theta = 2.6$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 292$ K
Prism, colourless
$0.35 \times 0.30 \times 0.25$ mm

Data collection

Rigaku SCXmini
    diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan
    (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.963$, $T_{max} = 0.976$

6256 measured reflections
2730 independent reflections
1540 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.174$
$S = 1.05$
2730 reflections
156 parameters
0 restraints
Primary atom site location: structure-invariant
    direct methods

Secondary atom site location: difference Fourier
    map
Hydrogen site location: inferred from
    neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.187P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ 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	$U_{iso}*/U_{eq}$
C1	0.2853 (4)	0.2569 (4)	0.9216 (2)	0.0526 (6)
C2	0.4367 (4)	0.3462 (3)	0.86392 (19)	0.0484 (6)
C3	0.6073 (4)	0.2769 (3)	0.8171 (2)	0.0531 (6)
C4	0.6958 (5)	0.0956 (4)	0.8063 (3)	0.0767 (9)
H4A	0.6878	0.0398	0.7300	0.115*
H4B	0.6210	0.0212	0.8473	0.115*
H4C	0.8359	0.1084	0.8346	0.115*
C5	0.8767 (4)	0.4291 (5)	0.7208 (2)	0.0715 (8)

H5A	0.9851	0.3738	0.7586	0.086*
H5B	0.9167	0.5557	0.7242	0.086*
C6	0.8532 (4)	0.3374 (4)	0.6016 (2)	0.0609 (7)
C7	0.6735 (6)	0.3212 (6)	0.5403 (3)	0.0988 (12)
H7	0.5557	0.3604	0.5729	0.119*
C8	0.6638 (8)	0.2462 (7)	0.4289 (3)	0.1207 (15)
H8	0.5399	0.2379	0.3872	0.145*
C9	0.8314 (11)	0.1859 (6)	0.3814 (3)	0.1187 (17)
H9	0.8239	0.1327	0.3073	0.142*
C10	1.0096 (9)	0.2029 (6)	0.4414 (5)	0.1221 (16)
H10	1.1271	0.1639	0.4083	0.146*
C11	1.0215 (5)	0.2769 (5)	0.5510 (3)	0.0872 (10)
H11	1.1467	0.2860	0.5916	0.105*
N1	0.4242 (3)	0.5221 (3)	0.85113 (17)	0.0557 (6)
N2	0.5786 (4)	0.5666 (3)	0.79918 (18)	0.0624 (6)
N3	0.6905 (3)	0.4179 (3)	0.77838 (16)	0.0567 (6)
O1	0.1510 (3)	0.3660 (2)	0.96493 (16)	0.0649 (6)
H1	0.0776	0.3125	1.0012	0.097*
O2	0.2875 (4)	0.0973 (3)	0.9257 (2)	0.0921 (8)
O1W	0.1065 (3)	0.7618 (2)	0.91803 (16)	0.0709 (6)
H1A	0.2137	0.6876	0.9038	0.106*
H1B	0.1686	0.8670	0.9279	0.106*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0594 (16)	0.0472 (15)	0.0502 (15)	−0.0021 (12)	0.0062 (12)	0.0056 (11)
C2	0.0559 (15)	0.0463 (13)	0.0418 (13)	−0.0018 (11)	0.0051 (11)	0.0043 (10)
C3	0.0562 (15)	0.0564 (16)	0.0425 (14)	−0.0037 (12)	0.0033 (11)	−0.0016 (11)
C4	0.079 (2)	0.0675 (19)	0.079 (2)	0.0093 (16)	0.0153 (16)	−0.0065 (16)
C5	0.0516 (16)	0.108 (2)	0.0536 (17)	−0.0118 (15)	0.0079 (13)	0.0128 (16)
C6	0.0641 (18)	0.0689 (18)	0.0531 (16)	0.0011 (14)	0.0143 (14)	0.0162 (13)
C7	0.080 (2)	0.148 (4)	0.059 (2)	0.008 (2)	−0.0022 (18)	−0.007 (2)
C8	0.142 (4)	0.149 (4)	0.062 (2)	−0.002 (3)	−0.012 (3)	0.003 (2)
C9	0.202 (6)	0.096 (3)	0.058 (2)	−0.009 (3)	0.048 (3)	0.003 (2)
C10	0.147 (4)	0.114 (3)	0.108 (4)	0.016 (3)	0.072 (3)	0.001 (3)
C11	0.083 (2)	0.092 (2)	0.090 (3)	0.0129 (19)	0.0349 (19)	0.013 (2)
N1	0.0634 (14)	0.0531 (13)	0.0527 (13)	−0.0007 (11)	0.0119 (10)	0.0125 (10)
N2	0.0677 (15)	0.0653 (15)	0.0570 (14)	−0.0054 (12)	0.0119 (11)	0.0172 (11)
N3	0.0560 (13)	0.0677 (15)	0.0446 (12)	−0.0065 (11)	0.0065 (10)	0.0052 (10)
O1	0.0683 (13)	0.0601 (11)	0.0732 (13)	0.0066 (10)	0.0273 (10)	0.0220 (9)
O2	0.1035 (17)	0.0458 (12)	0.134 (2)	−0.0006 (11)	0.0516 (15)	0.0183 (12)
O1W	0.0752 (13)	0.0540 (11)	0.0874 (14)	−0.0015 (10)	0.0318 (11)	0.0138 (10)

Geometric parameters (Å, º)

C1—O2	1.208 (3)	C6—C11	1.359 (4)
C1—O1	1.302 (3)	C7—C8	1.389 (5)

C1—C2	1.470 (3)	C7—H7	0.9300
C2—N1	1.360 (3)	C8—C9	1.338 (6)
C2—C3	1.372 (3)	C8—H8	0.9300
C3—N3	1.347 (3)	C9—C10	1.336 (6)
C3—C4	1.485 (4)	C9—H9	0.9300
C4—H4A	0.9600	C10—C11	1.366 (6)
C4—H4B	0.9600	C10—H10	0.9300
C4—H4C	0.9600	C11—H11	0.9300
C5—N3	1.463 (3)	N1—N2	1.301 (3)
C5—C6	1.509 (4)	N2—N3	1.353 (3)
C5—H5A	0.9700	O1—H1	0.8200
C5—H5B	0.9700	O1W—H1A	0.9192
C6—C7	1.352 (4)	O1W—H1B	0.8630
O2—C1—O1	124.7 (2)	C11—C6—C5	119.0 (3)
O2—C1—C2	122.0 (2)	C6—C7—C8	120.4 (4)
O1—C1—C2	113.3 (2)	C6—C7—H7	119.8
N1—C2—C3	109.0 (2)	C8—C7—H7	119.8
N1—C2—C1	122.0 (2)	C9—C8—C7	120.4 (4)
C3—C2—C1	129.0 (2)	C9—C8—H8	119.8
N3—C3—C2	103.5 (2)	C7—C8—H8	119.8
N3—C3—C4	123.9 (2)	C10—C9—C8	119.3 (4)
C2—C3—C4	132.6 (2)	C10—C9—H9	120.3
C3—C4—H4A	109.5	C8—C9—H9	120.3
C3—C4—H4B	109.5	C9—C10—C11	120.8 (4)
H4A—C4—H4B	109.5	C9—C10—H10	119.6
C3—C4—H4C	109.5	C11—C10—H10	119.6
H4A—C4—H4C	109.5	C6—C11—C10	121.0 (4)
H4B—C4—H4C	109.5	C6—C11—H11	119.5
N3—C5—C6	113.3 (2)	C10—C11—H11	119.5
N3—C5—H5A	108.9	N2—N1—C2	109.1 (2)
C6—C5—H5A	108.9	N1—N2—N3	106.8 (2)
N3—C5—H5B	108.9	C3—N3—N2	111.6 (2)
C6—C5—H5B	108.9	C3—N3—C5	129.6 (3)
H5A—C5—H5B	107.7	N2—N3—C5	118.8 (2)
C7—C6—C11	117.9 (3)	C1—O1—H1	109.5
C7—C6—C5	123.0 (3)	H1A—O1W—H1B	100.8

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1A···N1	0.92	1.96	2.870 (3)	172
O1W—H1B···O2[i]	0.86	1.88	2.734 (3)	171
O1—H1···O1W[ii]	0.82	1.75	2.563 (3)	168

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