

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

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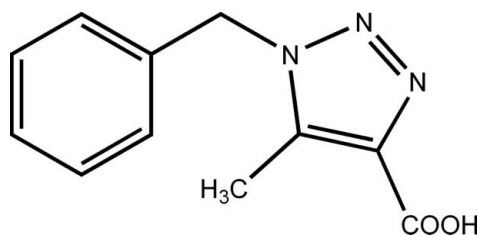
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.053; wR factor = 0.115; data-to-parameter ratio = 16.6.

In the title molecule, $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2$, the dihedral angle between the benzene and triazole rings is $76.47(10)^\circ$. The crystal structure exhibits intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds, which lead to the formation of helical chains along [001].

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: Xiao *et al.* (2008); Lin *et al.* (2008). For structural details of a monohydrate of the title compound, see: Zhao (2009).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2$
 $M_r = 217.23$
 Trigonal, $P3_1$
 $a = 10.1178(7)$ Å
 $c = 8.9971(8)$ Å
 $V = 797.64(11)$ Å³
 $Z = 3$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.18 \times 0.15$ mm

Data collection

 Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.921$, $T_{\max} = 1.000$

 8294 measured reflections
 2435 independent reflections
 1511 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.115$
 $S = 0.98$
 2435 reflections
 147 parameters

 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.13$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}^i$	0.82	1.91	2.721 (3)	171

 Symmetry code: (i) $-x + y + 1, -x + 1, z - \frac{1}{3}$.

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2044).

References

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supplementary materials

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

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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 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 (Fig. 1), a monohydrate of which has been previously reported (Zhao *et al.*, 2009).

The dihedral angle between the benzene and tirazole rings is $76.47(10)^\circ$. The crystal structure exhibits intermolecular O—H \cdots N hydrogen bonds which lead to the formation of one-dimensional chains along the [001] direction (Fig. 2.; Table 1).

Experimental

The title compound was prepared from azidomethylbenzene according to the reported method (El Khadem *et al.* 1968). NiCl₂ (1 mmol), NaN₃ (2 mmol) and the title compound (2 mmol) were placed in a thick Pyrex tube (*ca* 20 cm in length). After addition of 2.0 ml of water, the tube was frozen with liquid N₂, evacuated under vacuum, and sealed with a torch. The tube was heated at 120 °C for 3 days to give colourless prismatic crystals suitable for X-ray analysis.

Refinement

All H atoms were detected in a difference map, but were placed in calculated positions and refined using a riding motion approximation, with C—H=0.93–0.97Å and with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$. The bond distance O—H=0.82 Å and $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$.

Figures

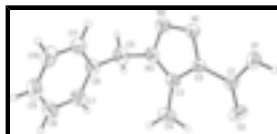


Fig. 1. The molecular structure of the title compound. The displacement ellipsoids are drawn at the 30% probability level.

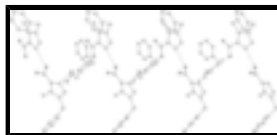


Fig. 2. Diagram of the molecules linked into one dimensional chains by O—H \cdots N hydrogen bonds.

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

Crystal data

$C_{11}H_{11}N_3O_2$	$D_x = 1.357 \text{ Mg m}^{-3}$
$M_r = 217.23$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Trigonal, $P3_1$	Cell parameters from 1614 reflections
Hall symbol: P 31	$\theta = 3.2\text{--}27.4^\circ$
$a = 10.1178 (7) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 8.9971 (8) \text{ \AA}$	$T = 293 \text{ K}$
$V = 797.64 (11) \text{ \AA}^3$	Prism, colourless
$Z = 3$	$0.20 \times 0.18 \times 0.15 \text{ mm}$
$F(000) = 342$	

Data collection

Rigaku SCXmini diffractometer	2435 independent reflections
Radiation source: fine-focus sealed tube graphite	1511 reflections with $I > 2\sigma(I)$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.049$
CCD_Profile_fitting scans	$\theta_{\text{max}} = 27.4^\circ$, $\theta_{\text{min}} = 3.3^\circ$
Absorption correction: multi-scan (<i>CrystalClear</i> ; Rigaku, 2005)	$h = -13 \rightarrow 12$
$T_{\text{min}} = 0.921$, $T_{\text{max}} = 1.000$	$k = -13 \rightarrow 13$
8294 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.053$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.115$	H-atom parameters constrained
$S = 0.98$	$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2]$
2435 reflections	where $P = (F_o^2 + 2F_c^2)/3$
147 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
1 restraint	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5374 (3)	0.4836 (3)	-0.0739 (3)	0.0501 (7)
C2	0.4072 (3)	0.4056 (3)	0.0273 (3)	0.0440 (6)
C3	0.2816 (3)	0.4222 (3)	0.0376 (3)	0.0505 (6)
C4	0.2356 (4)	0.5194 (4)	-0.0452 (4)	0.0751 (9)
H4A	0.1509	0.4566	-0.1088	0.113*
H4B	0.3198	0.5920	-0.1042	0.113*
H4C	0.2064	0.5725	0.0239	0.113*
C5	0.0466 (3)	0.2884 (4)	0.2051 (3)	0.0709 (9)
H5A	0.0400	0.3806	0.2120	0.085*
H5B	0.0333	0.2457	0.3042	0.085*
C6	-0.0779 (3)	0.1758 (3)	0.1057 (3)	0.0545 (7)
C7	-0.1626 (4)	0.2174 (4)	0.0197 (4)	0.0715 (9)
H7	-0.1439	0.3172	0.0231	0.086*
C8	-0.2762 (4)	0.1118 (5)	-0.0726 (4)	0.0880 (11)
H8	-0.3333	0.1408	-0.1309	0.106*
C9	-0.3039 (4)	-0.0348 (4)	-0.0778 (4)	0.0846 (11)
H9	-0.3792	-0.1056	-0.1405	0.102*
C10	-0.2215 (4)	-0.0772 (4)	0.0086 (4)	0.0809 (10)
H10	-0.2417	-0.1775	0.0060	0.097*
C11	-0.1093 (4)	0.0259 (4)	0.0992 (4)	0.0675 (8)
H11	-0.0531	-0.0045	0.1573	0.081*
N1	0.3952 (3)	0.3015 (3)	0.1301 (2)	0.0558 (6)
N2	0.2667 (3)	0.2531 (3)	0.2037 (2)	0.0650 (7)
N3	0.1987 (3)	0.3270 (3)	0.1477 (2)	0.0559 (6)
O1	0.6345 (2)	0.4335 (2)	-0.0625 (2)	0.0648 (6)
H1	0.7135	0.4916	-0.1068	0.097*
O2	0.5511 (2)	0.5822 (2)	-0.1573 (2)	0.0758 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0500 (16)	0.0416 (15)	0.0522 (16)	0.0180 (13)	-0.0017 (13)	-0.0012 (13)
C2	0.0462 (15)	0.0386 (13)	0.0462 (14)	0.0204 (12)	-0.0054 (12)	-0.0015 (11)
C3	0.0554 (16)	0.0502 (16)	0.0471 (14)	0.0273 (14)	-0.0109 (14)	-0.0081 (13)
C4	0.084 (2)	0.076 (2)	0.085 (2)	0.054 (2)	-0.0121 (19)	0.0013 (18)
C5	0.0624 (19)	0.102 (2)	0.0573 (18)	0.0480 (19)	0.0045 (14)	-0.0167 (17)
C6	0.0455 (16)	0.077 (2)	0.0447 (15)	0.0331 (15)	0.0101 (13)	-0.0023 (14)

supplementary materials

C7	0.059 (2)	0.076 (2)	0.088 (2)	0.0399 (19)	-0.0039 (18)	0.0008 (19)
C8	0.060 (2)	0.120 (3)	0.084 (2)	0.044 (2)	-0.0097 (18)	0.013 (2)
C9	0.056 (2)	0.096 (3)	0.078 (2)	0.019 (2)	0.0024 (17)	-0.012 (2)
C10	0.059 (2)	0.069 (2)	0.103 (3)	0.0232 (19)	0.018 (2)	-0.001 (2)
C11	0.061 (2)	0.079 (2)	0.070 (2)	0.0404 (18)	0.0098 (16)	0.0132 (18)
N1	0.0510 (14)	0.0652 (15)	0.0546 (14)	0.0315 (13)	0.0046 (11)	0.0118 (12)
N2	0.0598 (16)	0.087 (2)	0.0519 (14)	0.0393 (15)	0.0036 (12)	0.0153 (14)
N3	0.0499 (14)	0.0745 (16)	0.0479 (13)	0.0345 (13)	-0.0050 (11)	-0.0088 (12)
O1	0.0511 (11)	0.0661 (13)	0.0756 (14)	0.0281 (11)	0.0128 (10)	0.0121 (11)
O2	0.0828 (15)	0.0580 (13)	0.0785 (15)	0.0292 (11)	0.0135 (12)	0.0270 (12)

Geometric parameters (Å, °)

C1—O2	1.199 (3)	C6—C7	1.368 (4)
C1—O1	1.316 (3)	C6—C11	1.387 (4)
C1—C2	1.465 (4)	C7—C8	1.387 (5)
C2—N1	1.361 (3)	C7—H7	0.9300
C2—C3	1.366 (4)	C8—C9	1.366 (4)
C3—N3	1.343 (3)	C8—H8	0.9300
C3—C4	1.482 (4)	C9—C10	1.357 (5)
C4—H4A	0.9600	C9—H9	0.9300
C4—H4B	0.9600	C10—C11	1.363 (5)
C4—H4C	0.9600	C10—H10	0.9300
C5—N3	1.479 (3)	C11—H11	0.9300
C5—C6	1.500 (4)	N1—N2	1.315 (3)
C5—H5A	0.9700	N2—N3	1.342 (3)
C5—H5B	0.9700	O1—H1	0.8200
O2—C1—O1	124.8 (3)	C11—C6—C5	120.1 (3)
O2—C1—C2	122.4 (3)	C6—C7—C8	120.4 (3)
O1—C1—C2	112.8 (2)	C6—C7—H7	119.8
N1—C2—C3	108.7 (2)	C8—C7—H7	119.8
N1—C2—C1	123.2 (2)	C9—C8—C7	119.9 (3)
C3—C2—C1	128.1 (2)	C9—C8—H8	120.0
N3—C3—C2	104.3 (2)	C7—C8—H8	120.0
N3—C3—C4	123.8 (3)	C10—C9—C8	119.9 (4)
C2—C3—C4	131.9 (3)	C10—C9—H9	120.1
C3—C4—H4A	109.5	C8—C9—H9	120.1
C3—C4—H4B	109.5	C9—C10—C11	120.6 (4)
H4A—C4—H4B	109.5	C9—C10—H10	119.7
C3—C4—H4C	109.5	C11—C10—H10	119.7
H4A—C4—H4C	109.5	C10—C11—C6	120.7 (3)
H4B—C4—H4C	109.5	C10—C11—H11	119.7
N3—C5—C6	111.1 (2)	C6—C11—H11	119.7
N3—C5—H5A	109.4	N2—N1—C2	108.6 (2)
C6—C5—H5A	109.4	N1—N2—N3	106.9 (2)
N3—C5—H5B	109.4	N2—N3—C3	111.5 (2)
C6—C5—H5B	109.4	N2—N3—C5	118.5 (2)
H5A—C5—H5B	108.0	C3—N3—C5	129.9 (2)
C7—C6—C11	118.5 (3)	C1—O1—H1	109.5

C7—C6—C5	121.4 (3)		
O2—C1—C2—N1	-175.1 (3)	C9—C10—C11—C6	0.5 (5)
O1—C1—C2—N1	5.0 (4)	C7—C6—C11—C10	0.3 (4)
O2—C1—C2—C3	4.5 (4)	C5—C6—C11—C10	-179.6 (3)
O1—C1—C2—C3	-175.4 (3)	C3—C2—N1—N2	0.0 (3)
N1—C2—C3—N3	0.3 (3)	C1—C2—N1—N2	179.7 (2)
C1—C2—C3—N3	-179.4 (2)	C2—N1—N2—N3	-0.2 (3)
N1—C2—C3—C4	179.6 (3)	N1—N2—N3—C3	0.4 (3)
C1—C2—C3—C4	0.0 (5)	N1—N2—N3—C5	176.9 (2)
N3—C5—C6—C7	-109.3 (3)	C2—C3—N3—N2	-0.4 (3)
N3—C5—C6—C11	70.6 (3)	C4—C3—N3—N2	-179.9 (2)
C11—C6—C7—C8	-0.5 (5)	C2—C3—N3—C5	-176.4 (2)
C5—C6—C7—C8	179.3 (3)	C4—C3—N3—C5	4.2 (4)
C6—C7—C8—C9	0.0 (5)	C6—C5—N3—N2	-96.9 (3)
C7—C8—C9—C10	0.8 (5)	C6—C5—N3—C3	78.8 (4)
C8—C9—C10—C11	-1.0 (5)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...N1 ⁱ	0.82	1.91	2.721 (3)	171.

Symmetry codes: (i) $-x+y+1, -x+1, z-1/3$.

Fig. 1

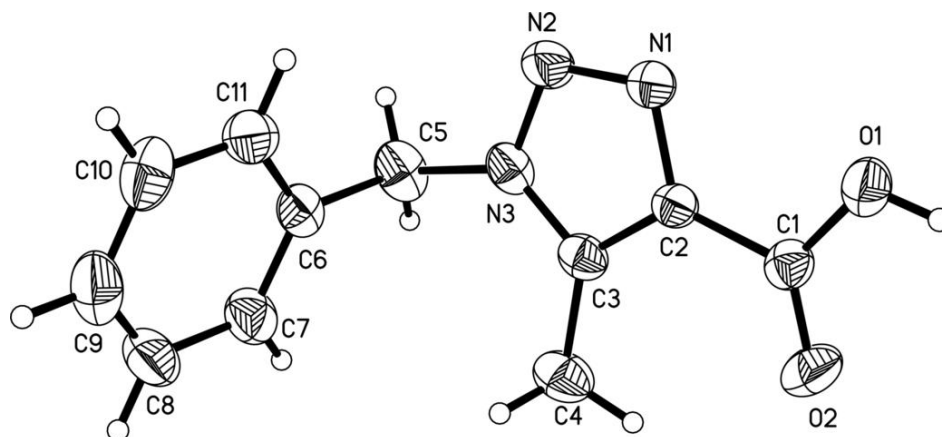


Fig. 2

