IUCrData

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9-Ethyl-6-methyl-7*H*-1,2,4-triazolo[4,3-*b*][1,2,4]-triazepin-8(9*H*)-one

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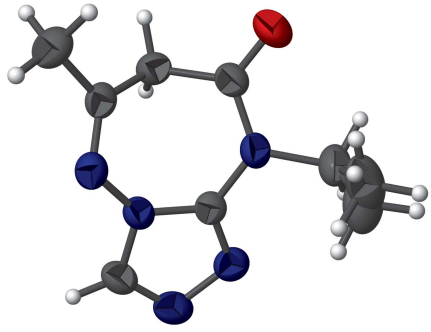
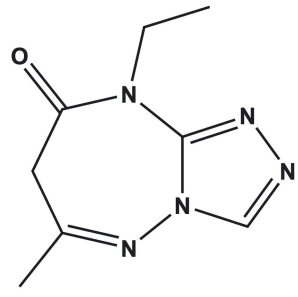
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_8H_{11}N_5O$, the triazepine ring displays a boat conformation. Its mean plane is inclined to the triazole ring by 22.10 (9)°. In the crystal, molecules are linked by C—H···O hydrogen bonds to form chains parallel to the *b*-axis direction. Inversion-related chains are linked *via* offset π–π interactions between the triazole rings, forming ribbons propagating in the *b*-axis direction. The terminal CH_3 group is disordered over two sets of sites, with a refined occupancy ratio of 0.48 (6):0.52 (6).

3D view



Chemical scheme



Structure description

1,2,4-Triazepine derivatives are useful in the treatment of HIV infections (Zhao *et al.*, 2005). It has been shown that heterocycles attached to a seven-membered ring possess important biological properties (Basile *et al.*, 1989; Gupta *et al.*, 2011). In a continuation of our studies on 1,2,4-triazolo[1,2,4]triazepine derivatives (Essassi *et al.*, 1977; Harmaoui *et al.*, 2015; Zemama *et al.*, 2009), we report herein on the synthesis and crystal structure of the title compound.

The molecule of the title compound, Fig. 1, is built up from a two fused rings with methyl and ethyl substituents. The triazepine ring (N1–N3/C1–C4) adopts a boat conformation, as indicated by the total puckering amplitude Q_T = 0.8176 (15) Å and the spherical polar angles θ_2 = 74.44 (11)° with φ_2 = −100.9 (2)° and φ_3 = −160.6 (4)°. The mean plane through the triazepine ring makes a dihedral angle of 22.10 (9)° with the triazole ring (N2/N4/N5/C4/C5).

In the crystal, molecules are linked by C5—H5···O1^{i} hydrogen bonds to form chains parallel to the *b* axis (Table 1 and Fig. 2). Inversion-related chains are linked by offset π–

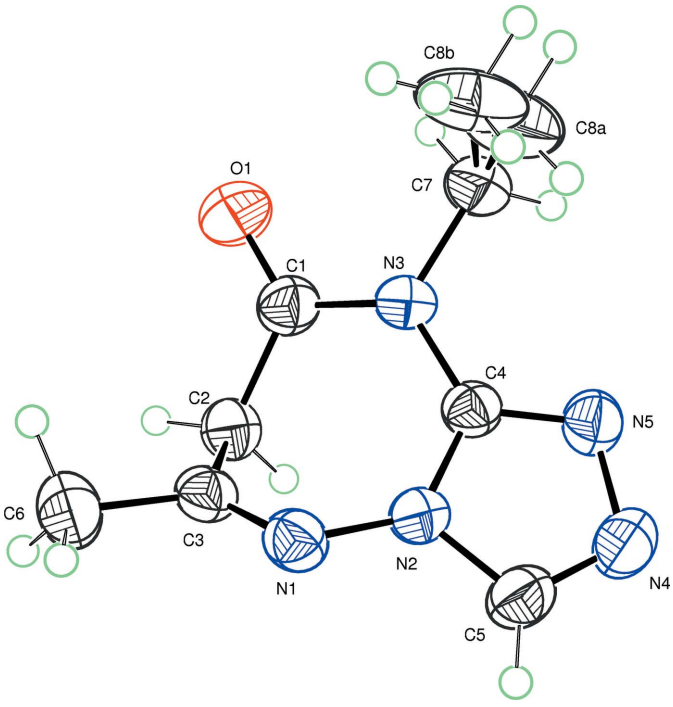


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

π interactions between triazole rings [$Cg\cdots Cg^{ii}$ = 3.581 (1) Å; *Cg* is the centroid of the N2/N4/N5/C4/C5 ring, interplanar distance = 3.150 (1) Å, slippage = 1.703 Å, symmetry code: (ii) $-x+1, -y+1, -z+2$], forming ribbons propagating in the *b*-axis direction (Fig. 2).

Synthesis and crystallization

To a solution of 6-methyl-7*H*-[1,2,4]triazolo[4,3-*b*][1,2,4] triazepin-8(9*H*)-one (1 g, 0.06 mol) in 30 ml of sodium methoxide (prepared from 30 ml of methanol and 0.15 g of sodium) was added 1 g (0.007 mol) of ethyl iodide, and the mixture was heated for 5 h. The solution was then concentrated to dryness under reduced pressure and the residue extracted with chloroform. The compound isolated was chromatographed on a silica column (eluent: chloroform/ethanol 95:5 *v*/*v*) and recrystallized from ethanol solution to give colourless crystals of the title compound (yield 70%).

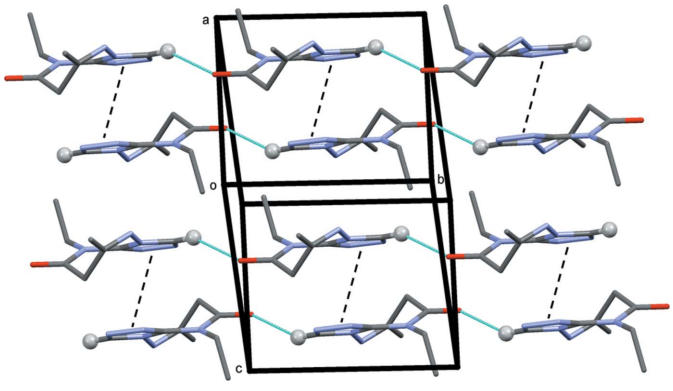


Figure 2
Crystal packing for the title compound, viewed normal to (101). The C—H···O hydrogen bonds (see Table 1) and π–π interactions are shown as cyan and black dashed lines, respectively.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.93	2.44	3.280 (2)	151

Symmetry code: (i) $x, y+1, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{11}N_5O$
M_r	193.22
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, *b*, *c* (Å)	7.8989 (3), 8.0880 (3), 8.2052 (3)
α, β, γ (°)	90.297 (2), 113.319 (2), 98.488 (2)
V (Å^3)	474.94 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.37 × 0.32 × 0.27
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.595, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14339, 2105, 1746
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.045, 0.133, 1.04
No. of reflections	2105
No. of parameters	137
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.17

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The terminal atom of the ethyl group (C8) is disordered over two sets of sites (C8*A*:C8*B*), with a refined occupancy ratio of 0.48 (6):0.52 (6).

Acknowledgements

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full crystallographic data

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Crystal data

$C_8H_{11}N_5O$
M_r = 193.22
Triclinic, $P\overline{1}$
a = 7.8989 (3) Å
b = 8.0880 (3) Å
c = 8.2052 (3) Å
α = 90.297 (2)°
β = 113.319 (2)°
γ = 98.488 (2)°
V = 474.94 (3) Å^3
Z = 2
$F(000)$ = 204
D_x = 1.351 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2105 reflections
θ = 2.6–27.1°
μ = 0.10 mm^{-1}
T = 296 K
Block, colourless
0.37 × 0.32 × 0.27 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.595, T_{max} = 0.747
14339 measured reflections
2105 independent reflections
1746 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.1°, θ_{min} = 2.6°
h = −10→10
k = −10→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.133
S = 1.04
2105 reflections
137 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.1549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.29573 (18)	0.49763 (16)	0.51599 (17)	0.0436 (3)	
N2	0.43326 (18)	0.52817 (15)	0.68976 (17)	0.0391 (3)	
N3	0.50084 (18)	0.25055 (16)	0.78018 (17)	0.0432 (3)	
N4	0.6626 (2)	0.67889 (18)	0.9111 (2)	0.0534 (4)	
N5	0.67342 (19)	0.51047 (17)	0.93918 (18)	0.0476 (3)	
O1	0.2994 (2)	0.00760 (15)	0.6764 (2)	0.0695 (4)	
C1	0.3253 (2)	0.15975 (19)	0.6984 (2)	0.0451 (4)	
C2	0.1666 (2)	0.2584 (2)	0.6363 (2)	0.0449 (4)	
H2A	0.0489	0.1818	0.5931	0.054*	
H2B	0.1728	0.3279	0.7358	0.054*	
C3	0.1734 (2)	0.36744 (19)	0.4908 (2)	0.0409 (3)	
C4	0.5351 (2)	0.42416 (18)	0.80585 (19)	0.0384 (3)	
C5	0.5207 (2)	0.6849 (2)	0.7627 (2)	0.0496 (4)	
H5	0.4836	0.7833	0.7128	0.059*	
C6	0.0294 (2)	0.3234 (2)	0.3070 (2)	0.0566 (5)	
H6A	0.0378	0.2065	0.2764	0.085*	
H6B	0.0458	0.4039	0.2286	0.085*	
H6C	−0.0949	0.3130	0.3069	0.085*	
C7	0.6598 (3)	0.1615 (2)	0.8760 (3)	0.0567 (5)	
H7A	0.7223	0.2094	0.9977	0.068*	
H7B	0.6107	0.0450	0.8796	0.068*	
C8A	0.790 (3)	0.168 (3)	0.802 (3)	0.086 (3)	0.48 (6)
H8A1	0.8876	0.1077	0.8717	0.129*	0.48 (6)
H8A2	0.7308	0.1178	0.6827	0.129*	0.48 (6)
H8A3	0.8425	0.2825	0.8010	0.129*	0.48 (6)
C8B	0.759 (3)	0.126 (3)	0.750 (4)	0.081 (4)	0.52 (6)
H8B1	0.8618	0.0680	0.8119	0.122*	0.52 (6)
H8B2	0.6713	0.0572	0.6464	0.122*	0.52 (6)
H8B3	0.8047	0.2297	0.7138	0.122*	0.52 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0447 (7)	0.0447 (7)	0.0404 (7)	0.0115 (6)	0.0146 (6)	0.0063 (5)
N2	0.0414 (7)	0.0349 (6)	0.0414 (7)	0.0077 (5)	0.0165 (5)	0.0026 (5)
N3	0.0437 (7)	0.0382 (7)	0.0468 (7)	0.0130 (5)	0.0150 (6)	0.0029 (5)
N4	0.0534 (8)	0.0454 (8)	0.0581 (9)	−0.0001 (6)	0.0219 (7)	−0.0071 (6)
N5	0.0443 (7)	0.0490 (8)	0.0459 (7)	0.0058 (6)	0.0151 (6)	−0.0025 (6)
O1	0.0705 (9)	0.0370 (7)	0.0891 (10)	0.0065 (6)	0.0204 (8)	0.0047 (6)

C1	0.0490 (9)	0.0383 (8)	0.0471 (8)	0.0063 (6)	0.0185 (7)	0.0056 (6)
C2	0.0391 (8)	0.0463 (9)	0.0504 (9)	0.0041 (6)	0.0201 (7)	0.0035 (7)
C3	0.0380 (7)	0.0432 (8)	0.0444 (8)	0.0131 (6)	0.0174 (6)	0.0026 (6)
C4	0.0393 (7)	0.0393 (8)	0.0399 (7)	0.0091 (6)	0.0183 (6)	0.0011 (6)
C5	0.0539 (10)	0.0363 (8)	0.0587 (10)	0.0045 (7)	0.0238 (8)	0.0010 (7)
C6	0.0470 (9)	0.0666 (12)	0.0486 (10)	0.0088 (8)	0.0112 (8)	0.0008 (8)
C7	0.0563 (11)	0.0512 (10)	0.0550 (10)	0.0203 (8)	0.0102 (8)	0.0034 (8)
C8A	0.090 (6)	0.097 (8)	0.091 (7)	0.052 (6)	0.044 (6)	0.017 (6)
C8B	0.089 (6)	0.075 (6)	0.123 (10)	0.046 (4)	0.076 (7)	0.039 (6)

Geometric parameters (Å, º)

N1—C3	1.275 (2)	C3—C6	1.487 (2)
N1—N2	1.4018 (18)	C5—H5	0.9300
N2—C5	1.360 (2)	C6—H6A	0.9954
N2—C4	1.3651 (19)	C6—H6B	0.9488
N3—C1	1.368 (2)	C6—H6C	0.9724
N3—C4	1.3888 (19)	C7—C8A	1.381 (17)
N3—C7	1.485 (2)	C7—C8B	1.58 (2)
N4—C5	1.295 (2)	C7—H7A	0.9700
N4—N5	1.392 (2)	C7—H7B	0.9700
N5—C4	1.303 (2)	C8A—H8A1	0.9600
O1—C1	1.218 (2)	C8A—H8A2	0.9600
C1—C2	1.503 (2)	C8A—H8A3	0.9600
C2—C3	1.501 (2)	C8B—H8B1	0.9600
C2—H2A	0.9700	C8B—H8B2	0.9600
C2—H2B	0.9700	C8B—H8B3	0.9600
C3—N1—N2	115.11 (13)	N2—C5—H5	124.5
C5—N2—C4	104.34 (13)	C3—C6—H6A	105.8
C5—N2—N1	122.76 (13)	C3—C6—H6B	110.7
C4—N2—N1	131.62 (12)	H6A—C6—H6B	115.5
C1—N3—C4	123.12 (13)	C3—C6—H6C	110.0
C1—N3—C7	119.04 (14)	H6A—C6—H6C	103.1
C4—N3—C7	116.85 (13)	H6B—C6—H6C	111.4
C5—N4—N5	107.22 (13)	C8A—C7—N3	114.7 (8)
C4—N5—N4	106.80 (13)	N3—C7—C8B	109.6 (7)
O1—C1—N3	121.73 (16)	C8A—C7—H7A	108.6
O1—C1—C2	122.02 (15)	N3—C7—H7A	108.6
N3—C1—C2	116.25 (13)	C8A—C7—H7B	108.6
C3—C2—C1	111.53 (13)	N3—C7—H7B	108.6
C3—C2—H2A	109.3	H7A—C7—H7B	107.6
C1—C2—H2A	109.3	C7—C8A—H8A1	109.5
C3—C2—H2B	109.3	C7—C8A—H8A2	109.5
C1—C2—H2B	109.3	H8A1—C8A—H8A2	109.5
H2A—C2—H2B	108.0	C7—C8A—H8A3	109.5
N1—C3—C6	117.64 (15)	H8A1—C8A—H8A3	109.5
N1—C3—C2	123.54 (14)	H8A2—C8A—H8A3	109.5

C6—C3—C2	118.82 (14)	C7—C8B—H8B1	109.5
N5—C4—N2	110.65 (13)	C7—C8B—H8B2	109.5
N5—C4—N3	124.87 (14)	H8B1—C8B—H8B2	109.5
N2—C4—N3	124.40 (13)	C7—C8B—H8B3	109.5
N4—C5—N2	110.98 (15)	H8B1—C8B—H8B3	109.5
N4—C5—H5	124.5	H8B2—C8B—H8B3	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.93	2.44	3.280 (2)	151

Symmetry code: (i) *x*, *y*+1, *z*.