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ISSN 2414-3146

4-Benzyl-1,2,4-triazaspiro[4.5]dec-1-ene-3-thione

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Received 7 December 2016
Accepted 9 December 2016

Edited by J. Simpson, University of Otago, New Zealand

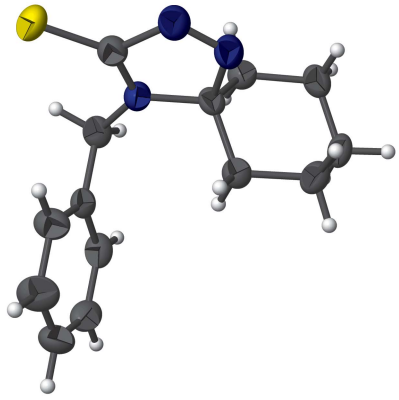
Keywords: crystal structure; triazole ring; cyclohexane ring; spiro-compounds; C—H···S hydrogen bonds.

CCDC reference: 1521677

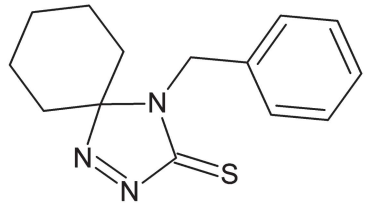
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{17}N_3S$, the cyclohexane ring adopts a chair conformation. The dihedral angle between the triazole and phenyl ring is 77.2 (3)°. In the crystal structure, C—H···S hydrogen link molecules into $C(7)$ chains along the b-axis direction. The crystal studied was refined as as an inversion twin.

3D view



Chemical scheme



Structure description

Heterocyclic 1,2,4-triazoline-5-thione derivatives exhibit a variety of biological properties including analgesic (Mekuskiene *et al.*, 1998), anti-inflammatory (Sahin *et al.*, 2001), bacteriostatic (Eweiss *et al.*, 1986) and antimitotic (Wujec *et al.*, 2004) activities. As part of our studies in this area, we determined the crystal structure of the title triazathione compound (Fig. 1).

The cyclohexane ring adopts a chair conformation with puckering parameters Q_T = 0.540 (5) Å, θ = 176.6 (5) ° and φ = 350 (9)°. The triazole ring is essentially planar (r.m.s. deviation = 0.004 Å) and makes a dihedral angle of 77.2 (3)° with the phenyl ring. The values of all geometric parameters are within normal ranges and comparable with the values for the related compound 4-allyl-1,2,4-triazaspiro[4.5]dec-1-ene-3-thione (Hassan *et al.*, 2016).

In the crystal, C10—H10···S1 hydrogen bonds link the molecules in a zigzag fashion into $C(7)$ chains along the b-axis direction (Table 1 and Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···S1^{i}	0.93	2.87	3.779 (6)	166

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

Synthesis and crystallization

The title compound was prepared according to our previously reported method (Hassan *et al.*, 2016). Colourless crystals suitable for X-ray diffraction were obtained from ethanol in 80% yield, using the slow evaporation method. M.p. 435–436 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was refined as as an inversion twin.

Acknowledgements

JPJ would like to acknowledge the NSF–MRI program (grant No. CHE-1039027) for funds to purchase the *X* ray diffractometer.

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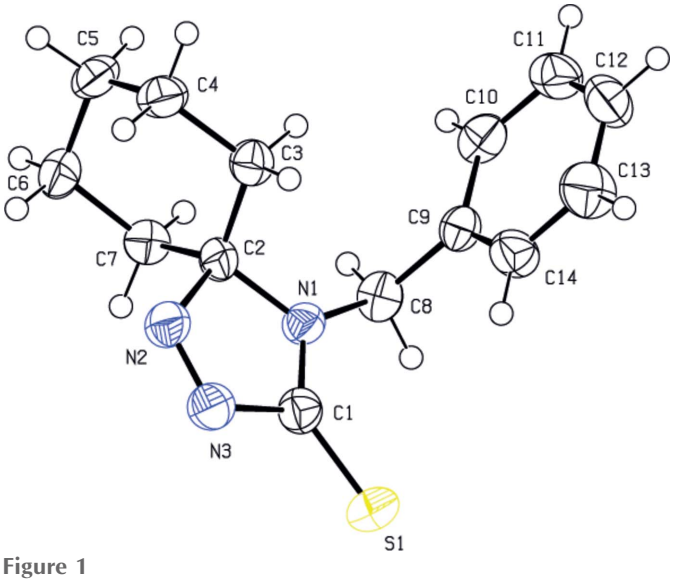

Figure 1
The title molecule, shown with 50% probability displacement ellipsoids.

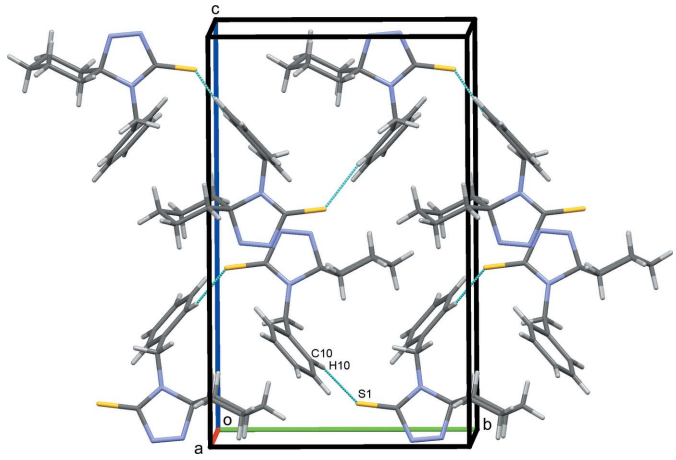

Figure 2
Packing of the title molecule, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{17}N_3S$
M_r	259.36
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	173
a, b, c (Å)	7.3730 (6), 10.7698 (7), 17.1056 (12)
V (Å^3)	1358.28 (17)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.99
Crystal size (mm)	0.34 × 0.32 × 0.22
Data collection	
Diffractometer	Rigaku Oxford Diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.531, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8805, 2607, 1974
R_{int}	0.071
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.138, 1.02
No. of reflections	2607
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.28
Absolute structure	Flack (1983), 1097 Friedel pairs; refined as an inversion twin
Absolute structure parameter	0.32 (4)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *WinGX* (Farrugia, 2012).

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full crystallographic data

IUCrData (2016). **1**, x161968 [https://doi.org/10.1107/S2414314616019684]

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4-Benzyl-1,2,4-triazaspiro[4.5]dec-1-ene-3-thione

Crystal data

$C_{14}H_{17}N_3S$
M_r = 259.36
Orthorhombic, $P2_12_12_1$
a = 7.3730 (6) Å
b = 10.7698 (7) Å
c = 17.1056 (12) Å
V = 1358.28 (17) Å^3
Z = 4
F(000) = 552
D_x = 1.268 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 1528 reflections
θ = 4.8–68.8°
μ = 1.99 mm^{-1}
T = 173 K
Irregular, colourless
0.34 × 0.32 × 0.22 mm

Data collection

Rigaku Oxford Diffraction diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.531, T_{max} = 1.000
8805 measured reflections
2607 independent reflections
1974 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 71.7°, θ_{min} = 4.9°
h = −9→8
k = −11→13
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.138
S = 1.02
2607 reflections
164 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 1097 Friedel pairs; refined as an inversion twin
Absolute structure parameter: 0.32 (4)

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.

Refinement. Refined as a two-component inversion twin

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5544 (7)	0.2039 (4)	0.4292 (2)	0.0379 (10)
C2	0.5495 (7)	0.4171 (4)	0.4180 (2)	0.0341 (9)
C3	0.7193 (7)	0.4963 (4)	0.4071 (3)	0.0384 (11)
H3A	0.823318	0.452406	0.428091	0.046*
H3B	0.740128	0.509365	0.351664	0.046*
C4	0.7017 (8)	0.6217 (5)	0.4477 (3)	0.0433 (13)
H4A	0.699621	0.609261	0.503813	0.052*
H4B	0.806647	0.672345	0.435208	0.052*
C5	0.5310 (7)	0.6895 (4)	0.4229 (3)	0.0446 (12)
H5A	0.521308	0.766687	0.451853	0.053*
H5B	0.538792	0.709806	0.367735	0.053*
C6	0.3633 (8)	0.6119 (5)	0.4372 (3)	0.0471 (13)
H6A	0.257830	0.656208	0.418029	0.056*
H6B	0.348178	0.599756	0.493036	0.056*
C7	0.3741 (7)	0.4850 (4)	0.3969 (3)	0.0405 (12)
H7A	0.271087	0.434823	0.412623	0.049*
H7B	0.368257	0.496254	0.340741	0.049*
C8	0.5758 (7)	0.2799 (4)	0.2940 (2)	0.0386 (11)
H8A	0.493986	0.337892	0.268686	0.046*
H8B	0.535983	0.196663	0.280833	0.046*
C9	0.7632 (7)	0.2991 (4)	0.2621 (3)	0.0356 (11)
C10	0.7936 (8)	0.3828 (4)	0.2015 (3)	0.0418 (12)
H10	0.696770	0.427247	0.180833	0.050*
C11	0.9654 (9)	0.4006 (5)	0.1718 (3)	0.0486 (14)
H11	0.983486	0.456565	0.131170	0.058*
C12	1.1088 (8)	0.3366 (5)	0.2017 (3)	0.0525 (15)
H12	1.224866	0.349381	0.182015	0.063*
C13	1.0808 (7)	0.2528 (5)	0.2612 (3)	0.0588 (15)
H13	1.178518	0.208316	0.281210	0.071*
C14	0.9102 (7)	0.2340 (5)	0.2915 (3)	0.0455 (12)
H14	0.893503	0.177266	0.331846	0.055*
N1	0.5623 (6)	0.2963 (3)	0.37830 (19)	0.0346 (8)
N2	0.5381 (6)	0.3770 (3)	0.5009 (2)	0.0423 (10)
N3	0.5407 (6)	0.2617 (4)	0.5075 (2)	0.0455 (10)
S1	0.5585 (2)	0.05329 (10)	0.41790 (8)	0.0495 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (2)	0.039 (2)	0.039 (2)	0.000 (2)	0.001 (3)	−0.0023 (19)
C2	0.040 (3)	0.034 (2)	0.028 (2)	0.0020 (19)	0.006 (2)	−0.0013 (16)
C3	0.040 (3)	0.039 (2)	0.036 (3)	0.000 (2)	0.001 (2)	−0.002 (2)
C4	0.057 (4)	0.034 (3)	0.039 (3)	−0.008 (2)	0.001 (2)	0.000 (2)
C5	0.062 (3)	0.026 (2)	0.045 (3)	0.000 (2)	0.003 (3)	−0.003 (2)
C6	0.053 (3)	0.037 (3)	0.051 (3)	0.011 (2)	0.010 (3)	−0.001 (2)

C7	0.043 (3)	0.038 (2)	0.041 (3)	−0.001 (2)	0.003 (2)	0.001 (2)
C8	0.039 (3)	0.043 (3)	0.033 (2)	0.002 (2)	−0.007 (2)	−0.0043 (19)
C9	0.041 (3)	0.034 (2)	0.032 (2)	−0.001 (2)	0.000 (2)	−0.0073 (19)
C10	0.056 (3)	0.037 (3)	0.033 (3)	0.006 (2)	−0.007 (2)	0.000 (2)
C11	0.067 (4)	0.048 (3)	0.031 (2)	−0.013 (3)	0.005 (3)	−0.001 (2)
C12	0.046 (4)	0.062 (4)	0.050 (3)	−0.007 (3)	0.014 (3)	−0.003 (3)
C13	0.036 (3)	0.062 (3)	0.079 (4)	0.010 (3)	0.000 (3)	0.013 (3)
C14	0.043 (3)	0.047 (3)	0.046 (3)	0.005 (2)	0.004 (2)	0.011 (2)
N1	0.041 (2)	0.0309 (18)	0.0317 (18)	−0.0008 (19)	0.0009 (19)	−0.0007 (14)
N2	0.058 (3)	0.037 (2)	0.0324 (19)	−0.001 (2)	0.005 (2)	−0.0029 (17)
N3	0.060 (3)	0.041 (2)	0.036 (2)	−0.002 (2)	0.006 (2)	−0.0008 (17)
S1	0.0572 (8)	0.0316 (6)	0.0597 (8)	−0.0043 (6)	0.0020 (8)	−0.0025 (6)

Geometric parameters (Å, º)

C1—N1	1.324 (5)	C7—H7A	0.9700
C1—N3	1.481 (5)	C7—H7B	0.9700
C1—S1	1.634 (4)	C8—N1	1.456 (5)
C2—N1	1.470 (5)	C8—C9	1.500 (7)
C2—N2	1.485 (5)	C8—H8A	0.9700
C2—C3	1.527 (7)	C8—H8B	0.9700
C2—C7	1.528 (7)	C9—C14	1.386 (7)
C3—C4	1.524 (6)	C9—C10	1.391 (6)
C3—H3A	0.9700	C10—C11	1.378 (8)
C3—H3B	0.9700	C10—H10	0.9300
C4—C5	1.516 (7)	C11—C12	1.362 (8)
C4—H4A	0.9700	C11—H11	0.9300
C4—H4B	0.9700	C12—C13	1.376 (7)
C5—C6	1.512 (7)	C12—H12	0.9300
C5—H5A	0.9700	C13—C14	1.375 (7)
C5—H5B	0.9700	C13—H13	0.9300
C6—C7	1.533 (6)	C14—H14	0.9300
C6—H6A	0.9700	N2—N3	1.247 (5)
C6—H6B	0.9700		
N1—C1—N3	106.4 (4)	C2—C7—H7A	109.4
N1—C1—S1	131.9 (3)	C6—C7—H7A	109.4
N3—C1—S1	121.7 (3)	C2—C7—H7B	109.4
N1—C2—N2	100.8 (3)	C6—C7—H7B	109.4
N1—C2—C3	112.6 (4)	H7A—C7—H7B	108.0
N2—C2—C3	109.0 (4)	N1—C8—C9	113.9 (4)
N1—C2—C7	111.6 (4)	N1—C8—H8A	108.8
N2—C2—C7	108.4 (4)	C9—C8—H8A	108.8
C3—C2—C7	113.4 (3)	N1—C8—H8B	108.8
C4—C3—C2	111.7 (4)	C9—C8—H8B	108.8
C4—C3—H3A	109.3	H8A—C8—H8B	107.7
C2—C3—H3A	109.3	C14—C9—C10	118.1 (5)
C4—C3—H3B	109.3	C14—C9—C8	121.3 (4)

C2—C3—H3B	109.3	C10—C9—C8	120.6 (4)
H3A—C3—H3B	107.9	C11—C10—C9	120.9 (5)
C5—C4—C3	111.7 (4)	C11—C10—H10	119.6
C5—C4—H4A	109.3	C9—C10—H10	119.6
C3—C4—H4A	109.3	C12—C11—C10	120.3 (5)
C5—C4—H4B	109.3	C12—C11—H11	119.9
C3—C4—H4B	109.3	C10—C11—H11	119.9
H4A—C4—H4B	107.9	C11—C12—C13	119.6 (5)
C6—C5—C4	111.5 (4)	C11—C12—H12	120.2
C6—C5—H5A	109.3	C13—C12—H12	120.2
C4—C5—H5A	109.3	C14—C13—C12	120.8 (5)
C6—C5—H5B	109.3	C14—C13—H13	119.6
C4—C5—H5B	109.3	C12—C13—H13	119.6
H5A—C5—H5B	108.0	C13—C14—C9	120.3 (5)
C5—C6—C7	112.2 (4)	C13—C14—H14	119.8
C5—C6—H6A	109.2	C9—C14—H14	119.8
C7—C6—H6A	109.2	C1—N1—C8	124.3 (4)
C5—C6—H6B	109.2	C1—N1—C2	111.0 (3)
C7—C6—H6B	109.2	C8—N1—C2	124.7 (3)
H6A—C6—H6B	107.9	N3—N2—C2	112.1 (3)
C2—C7—C6	111.4 (4)	N2—N3—C1	109.7 (4)
N1—C2—C3—C4	179.1 (4)	C8—C9—C14—C13	179.8 (5)
N2—C2—C3—C4	−69.9 (5)	N3—C1—N1—C8	−179.6 (4)
C7—C2—C3—C4	51.1 (5)	S1—C1—N1—C8	0.7 (9)
C2—C3—C4—C5	−53.4 (5)	N3—C1—N1—C2	−1.6 (6)
C3—C4—C5—C6	56.1 (5)	S1—C1—N1—C2	178.8 (4)
C4—C5—C6—C7	−55.7 (6)	C9—C8—N1—C1	−101.8 (6)
N1—C2—C7—C6	−178.9 (4)	C9—C8—N1—C2	80.4 (6)
N2—C2—C7—C6	71.0 (5)	N2—C2—N1—C1	1.5 (5)
C3—C2—C7—C6	−50.3 (5)	C3—C2—N1—C1	117.6 (5)
C5—C6—C7—C2	52.4 (6)	C7—C2—N1—C1	−113.4 (5)
N1—C8—C9—C14	54.4 (6)	N2—C2—N1—C8	179.6 (4)
N1—C8—C9—C10	−126.1 (4)	C3—C2—N1—C8	−64.4 (6)
C14—C9—C10—C11	−0.2 (7)	C7—C2—N1—C8	64.6 (6)
C8—C9—C10—C11	−179.8 (4)	N1—C2—N2—N3	−0.9 (5)
C9—C10—C11—C12	−0.3 (7)	C3—C2—N2—N3	−119.6 (5)
C10—C11—C12—C13	0.8 (8)	C7—C2—N2—N3	116.5 (5)
C11—C12—C13—C14	−0.8 (9)	C2—N2—N3—C1	0.0 (6)
C12—C13—C14—C9	0.3 (9)	N1—C1—N3—N2	1.0 (6)
C10—C9—C14—C13	0.2 (7)	S1—C1—N3—N2	−179.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···S1^{i}	0.93	2.87	3.779 (6)	166

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.