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4-Allyl-3-[(5-methyl-2-oxo-1,3-benzoxazol-3-yl)methyl]-1*H*-1,2,4-triazole-5(4*H*)-thione

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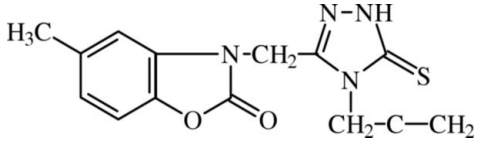
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.042; wR factor = 0.123; data-to-parameter ratio = 13.8.

In the molecular structure of the title compound, $C_{14}H_{14}N_4O_2S$, the benzoxazoline group is essentially planar, with a maximum deviation of 0.0358 (2) Å. N—H···S and C—H···S hydrogen bonds are primary interactions n the crystal structure. π–π Stacking interactions are present between the oxazoline ring systems, which are aligned in a parallel manner. The closest interaction has a perpendicular separation of 3.4 Å.

Related literature

The synthesis of the title compound was published by Salgın *et al.* (2007). The C—N bond distances and angles in the title compound are in agreement with values in our related structure (Köysal *et al.*, 2003). There are π–π stacking interactions between the five-membered rings N2/C10/N4/N3/C11 related by the symmetry code $(1-x, 1-y, 1-z)$.

For related literature, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{13}N_4O_2S$
M_r = 301.34
Orthorhombic, *Pbca*
a = 7.2848 (3) Å
b = 19.9139 (7) Å
c = 20.0634 (10) Å
V = 2910.6 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 (2) K
0.66 × 0.43 × 0.11 mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.881, T_{max} = 0.975
32373 measured reflections
2852 independent reflections
2153 reflections with $I > 2\sigma(I)$
R_{int} = 0.078

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.123
S = 1.00
2852 reflections
206 parameters
40 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—O2	1.198 (3)	N3—N4	1.373 (2)
C10—N4	1.299 (3)		
N1—C9—C10	111.70 (17)		
N1—C9—C10—N4	90.2 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···S1^{i}	0.87 (3)	2.41 (3)	3.284 (2)	179 (3)
C14A—H14B···S1^{ii}	0.93	2.74	3.506 (7)	140

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2265).

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supplementary materials

Acta Cryst. (2007). E**63**, o2462 [**doi:10.1107/S1600536807017163**]

4-Allyl-3-[(5-methyl-2-oxo-1,3-benzoxazol-3-yl)methyl]-1*H*-1,2,4-triazole-5(4*H*)-thione

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Comment

In the title compound, (I) (Fig. 1), the benzoxazoline ring system is essentially planar, with a maximum deviation from the mean plane of the nine-membered ring of -0.0358 (2) Å for atom C8. The C—N bond distances and angles are in agreement with values in our related structure, 3-[4-(2-chlorophenyl)piperazinomethyl]-5-methyl-1-benzoxazolin-2(3*H*)-one (Koysal *et al.*, 2003). The dihedral angle between the triazole and benzoxazoline ring systems is 68.62 (8)°. The anisotropic displacement parameters of allyl group atoms C13 and C14 are larger than those of the other atoms, suggesting that this group could be affected by high thermal motion.

The structure of compound (I) contains two intermolecular contacts, N3—H3···S1^{i} and C14*A*—H14*C*···S1^{ii} (Table 2). The adjacent S atom forms intermolecular hydrogen bonds linking the molecules into a three-dimensional network generating graph set $R^2_2(8)$ (Bernstein *et al.*, 1995).

π–π stacking interactions are present in (I), with the oxazoline ring systems aligned in a parallel manner. The closest interaction with a perpendicular seperation is 3.4 Å.

Experimental

The synthesis of the title compound and related derivatives was published by Salgın *et al.* (2007).

Refinement

In compound (I), atom C14 of the allyl group is disordered over two positions, with site occupancies of 0.64 (2) and 0.36 (2). The atomic displacement parameters are only slightly larger than those of the other atoms. The missing H atom in the allyl group could not be determined, either by fixing or by Fourier map, because disordered atom C14 atom is unexpectedly close to C13. N-bound atom N3 was located in a difference map and refined freely. The remaining H atoms were located geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic H, 0.97 Å for CH_2 and 0.96 Å for methyl H, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

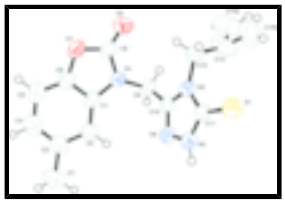

Fig. 1. The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

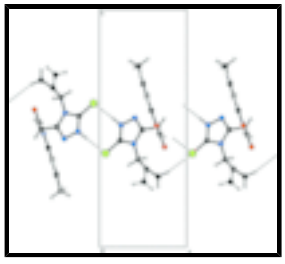

Fig. 2. A view of the C—H···S interactions between the molecules, down the *c* axis. Hydrogen bonds are indicated by dashed lines.

4-Allyl-3-[(5-methyl-2-oxo-1,3-benzoxazol-3-yl)methyl]-1*H*-1,2,4- triazole-5(4*H*)-thione

Crystal data

$C_{14}H_{13}N_4O_2S$	F_{000} = 1256
M_r = 301.34	D_x = 1.375 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 28896 reflections
a = 7.2848 (3) Å	θ = 2.0–27.2º
b = 19.9139 (7) Å	μ = 0.23 mm^{-1}
c = 20.0634 (10) Å	*T* = 293 (2) K
V = 2910.6 (2) Å^3	Prismatic plate, colourless
Z = 8	0.66 × 0.43 × 0.11 mm

Data collection

Stoe IPDS II diffractometer	2852 independent reflections
Radiation source: fine-focus sealed tube	2153 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.078
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 26.0º
T = 293(2) K	θ_{min} = 2.0º
ω scans	*h* = −8→8
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	*k* = −24→24
T_{min} = 0.881, T_{max} = 0.975	*l* = −24→24
32373 measured reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 0.4236P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.123	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.18 e Å^{-3}

2852 reflections

206 parameters

40 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0127 (14)

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
C1	0.5897 (3)	0.58195 (10)	0.72142 (10)	0.0474 (5)	
C2	0.5409 (3)	0.64148 (11)	0.69131 (12)	0.0546 (5)	
H2	0.5378	0.6456	0.6451	0.065*	
C3	0.4962 (3)	0.69562 (12)	0.73275 (13)	0.0618 (6)	
C4	0.5000 (4)	0.68683 (14)	0.80144 (14)	0.0700 (7)	
H4	0.4682	0.7229	0.8285	0.084*	
C5	0.5491 (3)	0.62663 (14)	0.83159 (13)	0.0666 (6)	
H5	0.5509	0.6216	0.8777	0.080*	
C6	0.5942 (3)	0.57553 (11)	0.78965 (11)	0.0527 (5)	
C7	0.4474 (4)	0.76210 (13)	0.70312 (19)	0.0875 (9)	
H7A	0.4061	0.7558	0.6581	0.105*	
H7B	0.5534	0.7907	0.7033	0.105*	
H7C	0.3514	0.7825	0.7289	0.105*	
C8	0.6892 (3)	0.47767 (11)	0.74739 (11)	0.0550 (5)	
C9	0.6716 (3)	0.50349 (11)	0.62625 (10)	0.0505 (5)	
H9A	0.7428	0.4625	0.6227	0.061*	
H9B	0.7398	0.5391	0.6045	0.061*	
C10	0.4916 (3)	0.49418 (10)	0.59153 (9)	0.0449 (5)	
C11	0.2362 (3)	0.44848 (11)	0.55338 (9)	0.0474 (5)	
C12	0.4464 (3)	0.36922 (11)	0.61302 (12)	0.0563 (5)	
H12A	0.3375	0.3457	0.6282	0.068*	
H12B	0.5283	0.3744	0.6508	0.068*	
C13	0.5381 (6)	0.32939 (16)	0.5608 (2)	0.1045 (12)	
C14A	0.6727 (11)	0.2975 (3)	0.5582 (4)	0.143 (3)	0.747 (11)

H14A	0.6702	0.2546	0.5400	0.171*	0.747 (11)
H14B	0.7823	0.3151	0.5743	0.171*	0.747 (11)
C14B	0.508 (3)	0.2757 (7)	0.5359 (8)	0.116 (8)	0.253 (11)
H14C	0.6033	0.2449	0.5306	0.140*	0.253 (11)
H14D	0.3903	0.2648	0.5219	0.140*	0.253 (11)
N1	0.6470 (2)	0.52012 (8)	0.69605 (8)	0.0487 (4)	
N2	0.3945 (2)	0.43549 (8)	0.58771 (8)	0.0454 (4)	
N3	0.2479 (3)	0.51378 (9)	0.53926 (8)	0.0484 (4)	
N4	0.4057 (2)	0.54327 (9)	0.56249 (8)	0.0484 (4)	
O1	0.6534 (2)	0.51119 (8)	0.80607 (7)	0.0610 (4)	
O2	0.7479 (3)	0.42155 (8)	0.74514 (9)	0.0727 (5)	
S1	0.06870 (8)	0.39441 (3)	0.53337 (3)	0.0651 (2)	
H3	0.164 (4)	0.5385 (13)	0.5201 (14)	0.074 (8)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0413 (10)	0.0534 (11)	0.0476 (11)	−0.0027 (8)	−0.0055 (8)	−0.0018 (9)
C2	0.0520 (12)	0.0544 (12)	0.0574 (13)	−0.0014 (9)	−0.0062 (10)	0.0018 (10)
C3	0.0502 (12)	0.0550 (12)	0.0802 (17)	0.0001 (10)	−0.0047 (12)	−0.0064 (11)
C4	0.0606 (14)	0.0706 (15)	0.0787 (17)	−0.0002 (12)	0.0043 (13)	−0.0218 (13)
C5	0.0648 (15)	0.0831 (17)	0.0519 (13)	−0.0045 (13)	−0.0002 (11)	−0.0119 (12)
C6	0.0480 (11)	0.0622 (13)	0.0480 (11)	−0.0032 (9)	−0.0053 (9)	−0.0011 (10)
C7	0.0822 (19)	0.0572 (15)	0.123 (3)	0.0068 (13)	−0.0054 (18)	−0.0013 (16)
C8	0.0544 (12)	0.0580 (13)	0.0525 (12)	−0.0011 (10)	−0.0115 (9)	0.0045 (10)
C9	0.0484 (11)	0.0576 (11)	0.0455 (10)	−0.0002 (9)	−0.0018 (9)	−0.0024 (9)
C10	0.0488 (11)	0.0485 (10)	0.0373 (9)	−0.0006 (8)	0.0032 (8)	−0.0013 (8)
C11	0.0509 (11)	0.0525 (11)	0.0389 (9)	0.0006 (9)	−0.0003 (8)	−0.0009 (8)
C12	0.0599 (13)	0.0515 (11)	0.0576 (13)	−0.0005 (10)	−0.0057 (10)	0.0097 (10)
C13	0.135 (3)	0.0572 (17)	0.121 (3)	0.0325 (19)	0.045 (2)	0.0157 (18)
C14A	0.133 (6)	0.090 (4)	0.205 (7)	0.012 (4)	0.068 (6)	0.026 (4)
C14B	0.109 (14)	0.119 (13)	0.121 (12)	0.049 (10)	−0.011 (9)	−0.052 (9)
N1	0.0518 (10)	0.0512 (9)	0.0431 (9)	0.0012 (7)	−0.0073 (8)	0.0017 (7)
N2	0.0500 (9)	0.0463 (9)	0.0398 (8)	0.0002 (7)	−0.0007 (7)	0.0005 (7)
N3	0.0505 (10)	0.0516 (10)	0.0431 (9)	0.0011 (8)	−0.0050 (8)	0.0001 (7)
N4	0.0518 (10)	0.0517 (10)	0.0418 (9)	−0.0025 (8)	−0.0016 (7)	−0.0005 (7)
O1	0.0697 (10)	0.0679 (10)	0.0453 (8)	−0.0009 (8)	−0.0112 (7)	0.0047 (7)
O2	0.0839 (12)	0.0609 (10)	0.0731 (11)	0.0134 (9)	−0.0164 (9)	0.0076 (8)
S1	0.0596 (4)	0.0580 (4)	0.0776 (4)	−0.0082 (3)	−0.0178 (3)	0.0040 (3)

Geometric parameters (Å, °)

C1—C6	1.375 (3)	C9—H9A	0.9700
C1—C2	1.377 (3)	C9—H9B	0.9700
C1—N1	1.396 (3)	C10—N4	1.299 (3)
C2—C3	1.400 (3)	C10—N2	1.368 (2)
C2—H2	0.9300	C11—N3	1.334 (3)
C3—C4	1.390 (4)	C11—N2	1.368 (2)
C3—C7	1.494 (4)	C11—S1	1.676 (2)

C4—C5	1.390 (4)	C12—N2	1.464 (3)
C4—H4	0.9300	C12—C13	1.474 (4)
C5—C6	1.361 (3)	C12—H12A	0.9700
C5—H5	0.9300	C12—H12B	0.9700
C6—O1	1.391 (3)	C13—C14A	1.169 (7)
C7—H7A	0.9600	C13—C14B	1.200 (13)
C7—H7B	0.9600	C14A—H14A	0.9300
C7—H7C	0.9600	C14A—H14B	0.9300
C8—O2	1.198 (3)	C14B—H14C	0.9300
C8—N1	1.368 (3)	C14B—H14D	0.9300
C8—O1	1.378 (3)	N3—N4	1.373 (2)
C9—N1	1.450 (3)	N3—H3	0.87 (3)
C9—C10	1.496 (3)		
C6—C1—C2	121.5 (2)	N4—C10—N2	111.65 (18)
C6—C1—N1	105.88 (18)	N4—C10—C9	122.53 (18)
C2—C1—N1	132.6 (2)	N2—C10—C9	125.80 (18)
C1—C2—C3	117.5 (2)	N3—C11—N2	103.75 (18)
C1—C2—H2	121.2	N3—C11—S1	128.44 (16)
C3—C2—H2	121.2	N2—C11—S1	127.80 (16)
C4—C3—C2	119.2 (2)	N2—C12—C13	110.8 (2)
C4—C3—C7	120.7 (2)	N2—C12—H12A	109.5
C2—C3—C7	120.1 (2)	C13—C12—H12A	109.5
C3—C4—C5	123.1 (2)	N2—C12—H12B	109.5
C3—C4—H4	118.5	C13—C12—H12B	109.5
C5—C4—H4	118.5	H12A—C12—H12B	108.1
C6—C5—C4	116.0 (2)	C14A—C13—C14B	69.5 (8)
C6—C5—H5	122.0	C14A—C13—C12	134.8 (6)
C4—C5—H5	122.0	C14B—C13—C12	133.9 (10)
C5—C6—C1	122.7 (2)	C13—C14A—H14A	120.0
C5—C6—O1	128.1 (2)	C13—C14A—H14B	120.0
C1—C6—O1	109.20 (19)	H14A—C14A—H14B	120.0
C3—C7—H7A	109.5	C13—C14B—H14C	120.0
C3—C7—H7B	109.5	C13—C14B—H14D	120.0
H7A—C7—H7B	109.5	H14C—C14B—H14D	120.0
C3—C7—H7C	109.5	C8—N1—C1	109.74 (17)
H7A—C7—H7C	109.5	C8—N1—C9	123.95 (18)
H7B—C7—H7C	109.5	C1—N1—C9	126.18 (17)
O2—C8—N1	129.0 (2)	C11—N2—C10	107.61 (16)
O2—C8—O1	123.5 (2)	C11—N2—C12	124.25 (17)
N1—C8—O1	107.55 (18)	C10—N2—C12	128.12 (17)
N1—C9—C10	111.70 (17)	C11—N3—N4	113.48 (17)
N1—C9—H9A	109.3	C11—N3—H3	126.7 (18)
C10—C9—H9A	109.3	N4—N3—H3	119.6 (18)
N1—C9—H9B	109.3	C10—N4—N3	103.51 (17)
C10—C9—H9B	109.3	C8—O1—C6	107.59 (16)
H9A—C9—H9B	107.9		
C6—C1—C2—C3	0.1 (3)	C6—C1—N1—C9	−177.32 (18)
N1—C1—C2—C3	−177.5 (2)	C2—C1—N1—C9	0.6 (4)

C1—C2—C3—C4	−1.0 (3)	C10—C9—N1—C8	110.1 (2)
C1—C2—C3—C7	178.2 (2)	C10—C9—N1—C1	−74.4 (3)
C2—C3—C4—C5	1.0 (4)	N3—C11—N2—C10	−0.4 (2)
C7—C3—C4—C5	−178.3 (2)	S1—C11—N2—C10	179.11 (16)
C3—C4—C5—C6	0.0 (4)	N3—C11—N2—C12	−178.78 (18)
C4—C5—C6—C1	−0.9 (4)	S1—C11—N2—C12	0.7 (3)
C4—C5—C6—O1	177.8 (2)	N4—C10—N2—C11	0.4 (2)
C2—C1—C6—C5	0.9 (3)	C9—C10—N2—C11	178.45 (18)
N1—C1—C6—C5	179.1 (2)	N4—C10—N2—C12	178.69 (18)
C2—C1—C6—O1	−178.09 (19)	C9—C10—N2—C12	−3.2 (3)
N1—C1—C6—O1	0.1 (2)	C13—C12—N2—C11	84.5 (3)
N1—C9—C10—N4	90.2 (2)	C13—C12—N2—C10	−93.5 (3)
N1—C9—C10—N2	−87.7 (2)	N2—C11—N3—N4	0.3 (2)
N2—C12—C13—C14A	131.5 (6)	S1—C11—N3—N4	−179.20 (15)
N2—C12—C13—C14B	−122.9 (12)	N2—C10—N4—N3	−0.2 (2)
O2—C8—N1—C1	−177.5 (2)	C9—C10—N4—N3	−178.34 (17)
O1—C8—N1—C1	2.0 (2)	C11—N3—N4—C10	−0.1 (2)
O2—C8—N1—C9	−1.4 (4)	O2—C8—O1—C6	177.7 (2)
O1—C8—N1—C9	178.11 (18)	N1—C8—O1—C6	−1.9 (2)
C6—C1—N1—C8	−1.3 (2)	C5—C6—O1—C8	−177.8 (2)
C2—C1—N1—C8	176.6 (2)	C1—C6—O1—C8	1.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···S1^{i}	0.87 (3)	2.41 (3)	3.284 (2)	179 (3)
C14A—H14B···S1^{ii}	0.93	2.74	3.506 (7)	140

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

Fig. 1

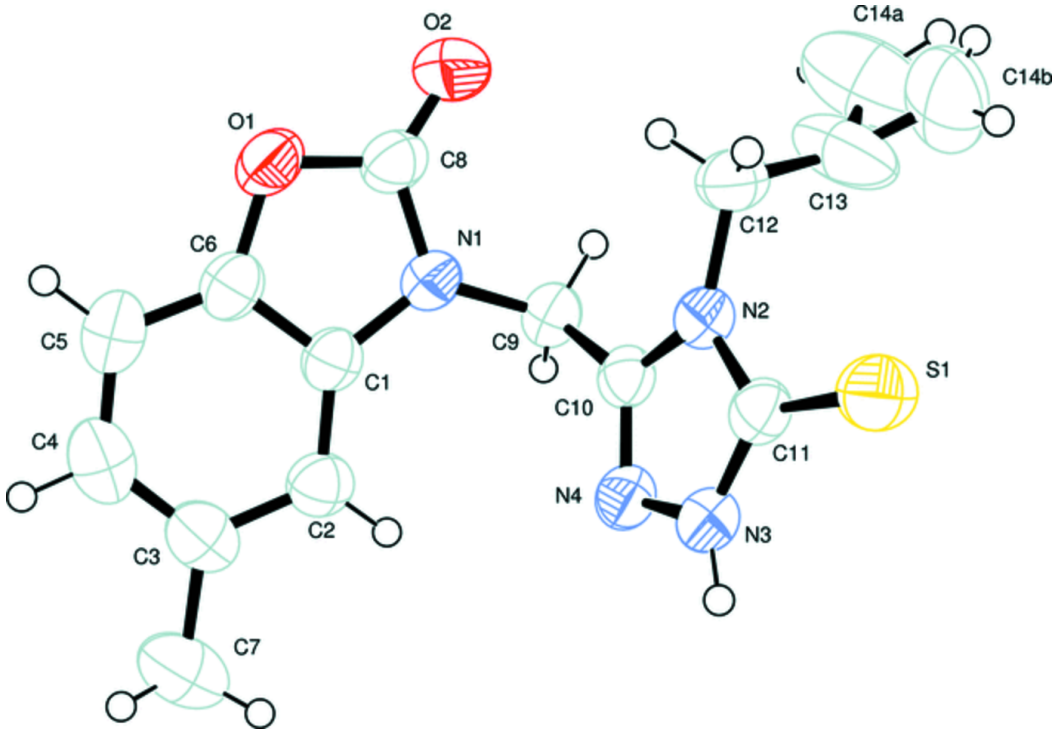
O2
O1
C8
C6
N1
C5
C1
C9
C4
C10
C2
C3
C7
C14a
C14b
C13
C12
N2
S1
C11
N4
N3

Fig. 2