

4-Amino-3-(1-naphthylloxymethyl)-1H-1,2,4-triazole-5(4H)-thione

Hoong-Kun Fun,^{a,*} Ching Kheng Quah,^{a,§} A. M. Vijesh,^{b,c} Shridhar Malladi^c and Arun M. Isloor^c

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bSeQuent Scientific Limited, No. 120 A & B, Industrial Area, Baikampady, New Mangalore, Karnataka 575 011, India, and ^cDepartment of Chemistry, National Institute of Technology-Karnataka, Surathkal, Mangalore 575 025, India

Correspondence e-mail: hkfun@usm.my

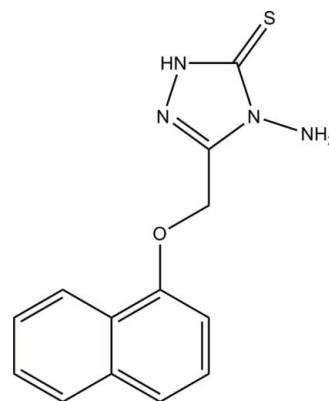
Received 26 November 2009; accepted 28 November 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.045; wR factor = 0.122; data-to-parameter ratio = 26.5.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{N}_4\text{OS}$, the dihedral angle between the triazole and naphthalene ring systems is $67.42(5)^\circ$. In the crystal, adjacent molecules are linked via two pairs of intermolecular $\text{N}-\text{H}\cdots\text{S}$ interactions, forming $R_2^2(8)$ and $R_2^2(10)$ ring motifs. Weak $\text{C}-\text{H}\cdots\text{S}$ interactions generate infinite chains along $[001]$ and the structure is further consolidated by $\text{C}-\text{H}\cdots\pi$ bonds and aromatic $\pi\cdots\pi$ stacking interactions [distance between the centroids of the triazole rings = $3.2479(7)$ Å].

Related literature

For general background to and the pharmacological activity of triazole derivatives, see: Amir *et al.* (2008); Sztanke *et al.* (2008); Kuş *et al.* (2008); Padmavathi *et al.* (2008); Isloor *et al.* (2009). For a related structure, see: Fun *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the preparation, see: Suresh (1992). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{N}_4\text{OS}$

$M_r = 272.33$

Monoclinic, $P2_1/c$

$a = 7.0023(1)$ Å

$b = 24.0785(4)$ Å

$c = 8.0915(1)$ Å

$\beta = 113.404(1)^\circ$

$V = 1252.02(3)$ Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.26$ mm⁻¹

$T = 100$ K

$0.38 \times 0.23 \times 0.07$ mm

Data collection

Bruker SMART APEXII CCD diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.908$, $T_{\max} = 0.983$

24326 measured reflections

5826 independent reflections

4223 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.122$

$S = 1.03$

5826 reflections

220 parameters

All H-atom parameters refined

$\Delta\rho_{\text{max}} = 0.51$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{S1}^i$	0.89 (2)	2.39 (2)	3.2857 (11)	176.2 (14)
$\text{N4}-\text{H1N4}\cdots\text{S1}^{ii}$	0.90 (2)	2.62 (2)	3.5075 (12)	167.3 (19)
$\text{C12}-\text{H12A}\cdots\text{S1}^{ii}$	0.96 (2)	2.836 (18)	3.5368 (13)	130.3 (12)
$\text{C9}-\text{H9A}\cdots\text{Cg1}^{iii}$	0.964 (17)	2.794 (18)	3.6345 (14)	146.8 (14)

Symmetry codes: (i) $-x, -y, -z + 1$; (ii) $-x, -y, -z$; (iii) $x, -y - \frac{3}{2}, z - \frac{3}{2}$. Cg1 is the centroid of C4-C8/C13 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HKF and CKQ thank Universiti Sains Malaysia (USM) for the Research University Golden Goose Grant (1001/PFIZIK/811012). CKQ thanks USM for a Research Fellowship. AMI is grateful to the Head of the Department of Chemistry and Director, NITK, Surathkal, India, for providing research facilities.

* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5255).

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supplementary materials

Acta Cryst. (2010). E66, o29-o30 [doi:10.1107/S1600536809051368]

4-Amino-3-(1-naphthylomethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

H.-K. Fun, C. K. Quah, A. M. Vijesh, S. Malladi and A. M. Isloor

Comment

1,2,4-triazole and its derivatives were reported to exhibit various pharmacological activities such as antimicrobial, analgesic, anti-inflammatory, anticancer and antioxidant properties (Amir *et al.*, 2008; Sztanke *et al.*, 2008; Kuş *et al.*, 2008; Padmavathi *et al.*, 2008). A few derivatives of triazoles have exhibited antimicrobial activity (Isloor *et al.*, 2009). Some of the present day drugs such as ribavirin (antiviral agent), rizatriptan (anti migraine agent), alprazolam (anxiolytic agent), fluconazole and itraconazole (antifungal agents) are the best examples for potent molecules possessing the triazole nucleus. The amino and mercapto groups of 1,2,4-triazoles serve as readily accessible nucleophilic centers of the preparation of N-bridged heterocycles. Keeping in view of the biological importance, we have synthesized the title compound to study its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. The triazole ring (C1/N1/N2/C2/N3) make an dihedral angle of 67.42 (5)° with naphthalene ring (C4-C13). Short intermolecular distances between the centroids of the triazole rings [3.2479 (7) Å] indicate the existence of $\pi\cdots\pi$ interactions. The molecular structure is linked *via* pairs of intermolecular N1—H1N1 \cdots S1 and N4—H1N4 \cdots S1 interactions, forming R^2_2 (8) and R^2_2 (10) ring motifs (Bernstein *et al.*, 1995), respectively. Bond lengths and angles are within normal ranges, and comparable to a closely related structure (Fun *et al.*, 2009). In the crystal packing (Fig. 2), the molecules are linked into infinite one-dimensional chains along the direction [0 0 1] *via* adjacent ring motifs and C12—H12A \cdots S1 interactions (Fig. 2). The crystal structure is further consolidated by C—H $\cdots\pi$ (Table 1) interactions.

Experimental

2-(1-Naphthyl)oxy)acetohydrazide (21.6 g, 1.00 mmol) was added slowly to a solution of potassium hydroxide (8.4 g, 1.50 mmol) in ethanol (150 ml). The resulting mixture was stirred well until a clear solution was obtained. Carbon disulphide (11.4 g, 1.50 mmol) was added drop-wise and the contents were stirred vigorously. Further stirring was continued for 24 h. The resulting mixture was diluted with ether (100 ml) and the precipitate formed was collected by filtration, washed with dry ether and dried at 65 %c under vacuum. It was used for the next step without any purification.

A mixture of the above synthesized potassium dithiocarbazinate (16.5 g, 0.50 mmol), hydrazine hydrate (99 %, 1.00 mmol) and water (2 ml) was heated gently to boil for 30 minutes. Heating was continued until the evacuation of hydrogen sulphide ceased. The reaction mixture was cooled to room temperature, diluted with water (100 ml) and acidified with HCl. The solid mass that separated was collected by filtration, washed with water and dried. Recrystallization was achieved from ethanol. The yield was 9.25 g (68 %), *m. p.* 470-471 K (Suresh, 1992).

Refinement

All H atoms were located in a difference Fourier map and refined freely.

Figures

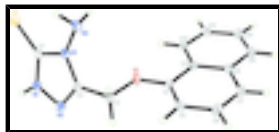


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids for non-H atoms.

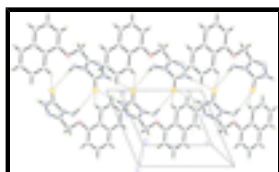


Fig. 2. The crystal structure of (I) viewed along the *b* axis. Intermolecular interactions are shown in dashed lines.

4-Amino-3-(1-naphthylloxymethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Crystal data

$C_{13}H_{12}N_4OS$

$M_r = 272.33$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.0023$ (1) Å

$b = 24.0785$ (4) Å

$c = 8.0915$ (1) Å

$\beta = 113.404$ (1)°

$V = 1252.02$ (3) Å³

$Z = 4$

$F(000) = 568$

$D_x = 1.445$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5692 reflections

$\theta = 2.9$ – 35.1 °

$\mu = 0.26$ mm⁻¹

$T = 100$ K

Plate, yellow

$0.38 \times 0.23 \times 0.07$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)

$T_{\min} = 0.908$, $T_{\max} = 0.983$

24326 measured reflections

5826 independent reflections

4223 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 35.8$ °, $\theta_{\min} = 2.9$ °

$h = -11 \rightarrow 11$

$k = -39 \rightarrow 38$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.122$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.214P]$
5826 reflections	where $P = (F_o^2 + 2F_c^2)/3$
220 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.00629 (5)	0.044619 (13)	0.25791 (4)	0.01810 (8)
O1	0.52129 (13)	-0.11362 (4)	0.14188 (11)	0.01666 (16)
N1	0.22651 (15)	-0.04341 (4)	0.46060 (13)	0.01481 (17)
N2	0.38889 (16)	-0.07854 (4)	0.47914 (13)	0.01636 (18)
N3	0.33179 (15)	-0.01814 (4)	0.25913 (12)	0.01411 (17)
N4	0.37675 (18)	0.01243 (5)	0.13172 (14)	0.0193 (2)
C1	0.18602 (17)	-0.00623 (5)	0.32721 (14)	0.01419 (19)
C2	0.44981 (17)	-0.06189 (5)	0.35421 (15)	0.01418 (19)
C3	0.61978 (18)	-0.08762 (5)	0.31417 (15)	0.0162 (2)
C4	0.65096 (17)	-0.13872 (5)	0.07391 (14)	0.01338 (19)
C5	0.86351 (18)	-0.14187 (5)	0.16169 (16)	0.0165 (2)
C6	0.98108 (19)	-0.16981 (5)	0.07908 (17)	0.0195 (2)
C7	0.88492 (19)	-0.19351 (5)	-0.08729 (17)	0.0187 (2)
C8	0.66644 (18)	-0.19040 (5)	-0.18076 (15)	0.01484 (19)
C9	0.5615 (2)	-0.21540 (5)	-0.35253 (16)	0.0185 (2)
C10	0.3499 (2)	-0.21323 (5)	-0.43817 (16)	0.0199 (2)
C11	0.2304 (2)	-0.18580 (5)	-0.35758 (16)	0.0178 (2)
C12	0.32576 (18)	-0.16061 (5)	-0.19258 (15)	0.01452 (19)
C13	0.54499 (17)	-0.16261 (4)	-0.10061 (14)	0.01255 (18)
H3A	0.693 (2)	-0.1148 (6)	0.405 (2)	0.013 (3)*
H3B	0.715 (2)	-0.0602 (6)	0.310 (2)	0.015 (4)*
H5A	0.940 (3)	-0.1254 (7)	0.279 (2)	0.022 (4)*
H6A	1.131 (3)	-0.1723 (7)	0.134 (2)	0.029 (4)*
H7A	0.962 (3)	-0.2110 (7)	-0.143 (2)	0.026 (4)*

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H9A	0.647 (3)	-0.2335 (7)	-0.404 (2)	0.031 (5)*
H10A	0.288 (3)	-0.2318 (7)	-0.550 (2)	0.028 (4)*
H11A	0.080 (3)	-0.1865 (7)	-0.414 (2)	0.023 (4)*
H12A	0.240 (3)	-0.1418 (7)	-0.142 (2)	0.022 (4)*
H1N1	0.162 (3)	-0.0455 (7)	0.536 (3)	0.031 (5)*
H1N4	0.284 (3)	0.0023 (8)	0.022 (3)	0.035 (5)*
H2N4	0.354 (3)	0.0475 (8)	0.153 (3)	0.035 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01841 (14)	0.02112 (15)	0.01629 (13)	0.00540 (10)	0.00852 (11)	0.00193 (10)
O1	0.0150 (4)	0.0207 (4)	0.0148 (3)	0.0010 (3)	0.0065 (3)	-0.0072 (3)
N1	0.0161 (4)	0.0159 (4)	0.0151 (4)	0.0006 (3)	0.0090 (4)	-0.0001 (3)
N2	0.0187 (4)	0.0159 (4)	0.0170 (4)	0.0015 (4)	0.0098 (4)	-0.0006 (3)
N3	0.0157 (4)	0.0156 (4)	0.0129 (4)	0.0004 (3)	0.0077 (3)	-0.0001 (3)
N4	0.0248 (5)	0.0216 (5)	0.0153 (4)	0.0013 (4)	0.0122 (4)	0.0031 (4)
C1	0.0144 (5)	0.0157 (5)	0.0133 (4)	-0.0014 (4)	0.0064 (4)	-0.0022 (3)
C2	0.0151 (5)	0.0143 (5)	0.0138 (4)	-0.0005 (4)	0.0064 (4)	-0.0029 (3)
C3	0.0157 (5)	0.0191 (5)	0.0135 (4)	0.0012 (4)	0.0056 (4)	-0.0044 (4)
C4	0.0141 (4)	0.0139 (5)	0.0140 (4)	0.0013 (3)	0.0076 (4)	-0.0015 (3)
C5	0.0148 (5)	0.0188 (5)	0.0156 (5)	0.0000 (4)	0.0057 (4)	-0.0023 (4)
C6	0.0145 (5)	0.0233 (6)	0.0218 (5)	0.0027 (4)	0.0085 (4)	-0.0006 (4)
C7	0.0187 (5)	0.0193 (5)	0.0218 (5)	0.0035 (4)	0.0118 (5)	-0.0007 (4)
C8	0.0187 (5)	0.0130 (5)	0.0159 (4)	0.0008 (4)	0.0100 (4)	0.0001 (4)
C9	0.0259 (6)	0.0157 (5)	0.0172 (5)	-0.0006 (4)	0.0120 (5)	-0.0034 (4)
C10	0.0261 (6)	0.0174 (5)	0.0153 (5)	-0.0032 (4)	0.0072 (5)	-0.0039 (4)
C11	0.0185 (5)	0.0177 (5)	0.0159 (5)	-0.0026 (4)	0.0054 (4)	0.0000 (4)
C12	0.0156 (5)	0.0137 (5)	0.0148 (4)	-0.0003 (4)	0.0066 (4)	-0.0003 (4)
C13	0.0145 (5)	0.0111 (4)	0.0129 (4)	0.0004 (3)	0.0065 (4)	-0.0003 (3)

Geometric parameters (\AA , $^\circ$)

S1—C1	1.6842 (12)	C5—C6	1.4192 (16)
O1—C4	1.3743 (12)	C5—H5A	0.971 (17)
O1—C3	1.4308 (13)	C6—C7	1.3678 (18)
N1—C1	1.3431 (14)	C6—H6A	0.964 (18)
N1—N2	1.3768 (13)	C7—C8	1.4138 (17)
N1—H1N1	0.894 (19)	C7—H7A	0.931 (17)
N2—C2	1.3066 (14)	C8—C9	1.4223 (16)
N3—C1	1.3691 (13)	C8—C13	1.4246 (15)
N3—C2	1.3710 (15)	C9—C10	1.3648 (19)
N3—N4	1.4005 (13)	C9—H9A	0.964 (17)
N4—H1N4	0.90 (2)	C10—C11	1.4123 (17)
N4—H2N4	0.888 (19)	C10—H10A	0.947 (18)
C2—C3	1.4872 (15)	C11—C12	1.3740 (16)
C3—H3A	0.966 (15)	C11—H11A	0.967 (17)
C3—H3B	0.948 (15)	C12—C13	1.4160 (16)
C4—C5	1.3732 (16)	C12—H12A	0.960 (16)

C4—C13	1.4294 (15)		
C4—O1—C3	116.29 (9)	C4—C5—H5A	123.0 (10)
C1—N1—N2	113.50 (9)	C6—C5—H5A	117.3 (10)
C1—N1—H1N1	125.7 (12)	C7—C6—C5	120.69 (11)
N2—N1—H1N1	120.7 (12)	C7—C6—H6A	116.6 (10)
C2—N2—N1	103.67 (9)	C5—C6—H6A	122.6 (10)
C1—N3—C2	108.34 (9)	C6—C7—C8	120.62 (10)
C1—N3—N4	127.42 (10)	C6—C7—H7A	121.0 (11)
C2—N3—N4	123.73 (9)	C8—C7—H7A	118.4 (11)
N3—N4—H1N4	107.3 (12)	C7—C8—C9	121.97 (10)
N3—N4—H2N4	104.3 (12)	C7—C8—C13	119.74 (10)
H1N4—N4—H2N4	109.4 (18)	C9—C8—C13	118.27 (10)
N1—C1—N3	103.33 (9)	C10—C9—C8	121.01 (10)
N1—C1—S1	130.05 (8)	C10—C9—H9A	122.4 (11)
N3—C1—S1	126.60 (9)	C8—C9—H9A	116.6 (11)
N2—C2—N3	111.15 (10)	C9—C10—C11	120.41 (11)
N2—C2—C3	125.18 (11)	C9—C10—H10A	117.4 (10)
N3—C2—C3	123.64 (10)	C11—C10—H10A	122.2 (10)
O1—C3—C2	106.07 (9)	C12—C11—C10	120.45 (11)
O1—C3—H3A	110.6 (9)	C12—C11—H11A	119.1 (10)
C2—C3—H3A	109.9 (8)	C10—C11—H11A	120.4 (10)
O1—C3—H3B	110.0 (9)	C11—C12—C13	120.17 (10)
C2—C3—H3B	110.6 (9)	C11—C12—H12A	118.5 (10)
H3A—C3—H3B	109.6 (13)	C13—C12—H12A	121.4 (10)
C5—C4—O1	124.75 (10)	C12—C13—C8	119.68 (10)
C5—C4—C13	121.29 (9)	C12—C13—C4	122.31 (9)
O1—C4—C13	113.96 (9)	C8—C13—C4	117.98 (10)
C4—C5—C6	119.66 (11)		
C1—N1—N2—C2	0.50 (13)	C4—C5—C6—C7	0.18 (19)
N2—N1—C1—N3	-0.61 (12)	C5—C6—C7—C8	0.32 (19)
N2—N1—C1—S1	-179.18 (9)	C6—C7—C8—C9	-178.92 (12)
C2—N3—C1—N1	0.48 (12)	C6—C7—C8—C13	-0.36 (18)
N4—N3—C1—N1	-171.51 (10)	C7—C8—C9—C10	178.15 (11)
C2—N3—C1—S1	179.11 (9)	C13—C8—C9—C10	-0.43 (17)
N4—N3—C1—S1	7.12 (17)	C8—C9—C10—C11	0.18 (18)
N1—N2—C2—N3	-0.17 (12)	C9—C10—C11—C12	0.48 (18)
N1—N2—C2—C3	-178.26 (11)	C10—C11—C12—C13	-0.87 (17)
C1—N3—C2—N2	-0.20 (13)	C11—C12—C13—C8	0.61 (16)
N4—N3—C2—N2	172.15 (10)	C11—C12—C13—C4	-177.82 (11)
C1—N3—C2—C3	177.92 (10)	C7—C8—C13—C12	-178.58 (11)
N4—N3—C2—C3	-9.73 (17)	C9—C8—C13—C12	0.03 (16)
C4—O1—C3—C2	177.76 (9)	C7—C8—C13—C4	-0.08 (16)
N2—C2—C3—O1	110.95 (12)	C9—C8—C13—C4	178.53 (10)
N3—C2—C3—O1	-66.91 (14)	C5—C4—C13—C12	179.03 (11)
C3—O1—C4—C5	2.21 (16)	O1—C4—C13—C12	-0.50 (15)
C3—O1—C4—C13	-178.28 (9)	C5—C4—C13—C8	0.57 (16)
O1—C4—C5—C6	178.85 (11)	O1—C4—C13—C8	-178.96 (9)
C13—C4—C5—C6	-0.63 (17)		

supplementary materials

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>
N1—H1N1···S1 ⁱ	0.89 (2)	2.39 (2)	3.2857 (11)	176.2 (14)
N4—H1N4···S1 ⁱⁱ	0.90 (2)	2.62 (2)	3.5075 (12)	167.3 (19)
C12—H12A···S1 ⁱⁱ	0.96 (2)	2.836 (18)	3.5368 (13)	130.3 (12)
C9—H9A···Cg1 ⁱⁱⁱ	0.964 (17)	2.794 (18)	3.6345 (14)	146.8 (14)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x, -y, -z$; (iii) $x, -y-3/2, z-3/2$.

Fig. 1

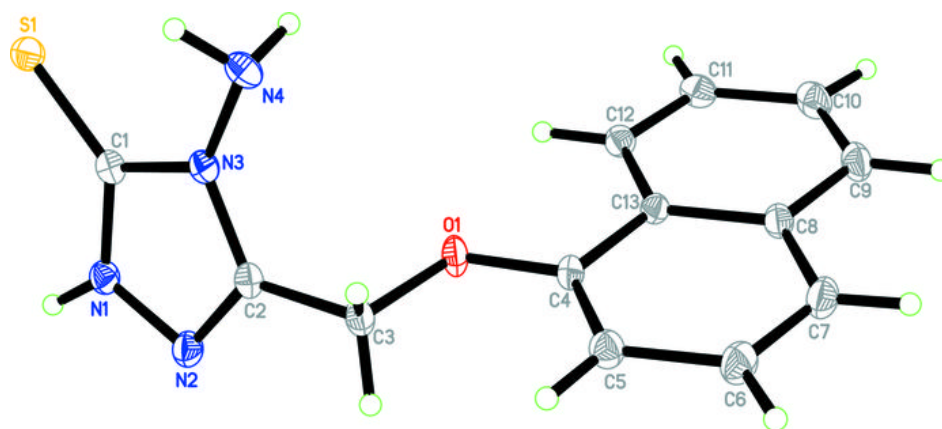


Fig. 2

