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3-(4-Fluorobenzyl)-4-(2-methoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Raja Ansar Hussain Khan,[a] Rashid Iqbal,[a] Habib-ur-Rehman,[a] Ghulam Qadeer[b]* and Wai-Yeung Wong[c]

[a]Department of Chemistry, University of Azad Jammu and Kashmir, Kashmir, Pakistan, [b]Department of Chemistry, Quaid-i-Azam Univeristy, Islamabad 45320, Pakistan, and [c]Department of Chemistry, Hong Kong Baptist University, Waterloo Road, Kowloon Tong, Hong Kong
Correspondence e-mail: qadeerqau@yahoo.com

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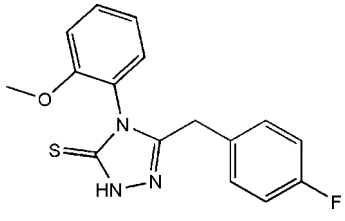
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.091; data-to-parameter ratio = 13.5.

The title compound, $C_{16}H_{14}FN_3OS$, is an important biologically active heterocyclic compound, containing one planar five-membered and two planar six-membered rings. The five-membered ring is oriented with respect to the six-membered rings at dihedral angles of 86.96 (3) and 74.42 (3)°. In the crystal structure, intermolecular N—H···S and C—H···F hydrogen bonds link the molecules into infinite chains.

Related literature

For bond-length data, see: Allen *et al.* (1987). For general background, see: Holla *et al.* (1998); Turan-Zitouni *et al.* (1999); Demirbas *et al.* (2002); Paulvannan *et al.* (2000); Kritsanida *et al.* (2002); Omar *et al.* (1986). For related structures, see: Öztürk *et al.* (2004*a*,*b*); Zhang *et al.* (2004).



Experimental

Crystal data

$C_{16}H_{14}FN_3OS$
M_r = 315.36
Monoclinic, $P2_1/n$
a = 7.4910 (7) Å
b = 16.1656 (15) Å
c = 12.9147 (12) Å
β = 91.956 (2)°
V = 1563.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 298 (2) K
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area-dedector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.873, T_{max} = 0.950
7491 measured reflections
2735 independent reflections
2209 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.02
2735 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···S1^{i}	0.824 (19)	2.465 (17)	3.2789 (16)	171.1 (16)
C5—H5A···F1^{ii}	0.93	2.37	3.186 (3)	146

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2291).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Demirbas, N., Ugurluoglu, R. & Demirbas, A. (2002). *Bioorg. Med. Chem.* **10**, 3717–3723.
Holla, B. S., Gonsalves, R. & Shenoy, S. (1998). *Farmaco*, **53**, 574–578.
Kritsanida, M., Mouroutsou, A., Marakos, P., Pouli, N., Papakonstantinou-Garoufalias, S., Pannecouque, C., Witvrouw, M. & Clercq, E. D. (2002). *Farmaco*, **57**, 253–257.
Omar, A., Mohsen, M. E. & Wafa, O. A. (1986). *Heterocycl. Chem.* **23**, 1339–1341.
Öztürk, S., Akkurt, M., Cansız, A., Koparır, M., Şekerci, M. & Heinemann, F. W. (2004*a*). *Acta Cryst.* E**60**, o425–o427.
Öztürk, S., Akkurt, M., Cansız, A., Koparır, M., Şekerci, M. & Heinemann, F. W. (2004*b*). *Acta Cryst.* E**60**, o642–o644.
Paulvannan, K., Chen, T. & Hale, R. (2000). *Tetrahedron*, **56**, 8071–8076.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Turan-Zitouni, G., Kaplancikli, Z. A., Erol, K. & Kilic, F. S. (1999). *Farmaco*, **54**, 218–223.
Zhang, L.-X., Zhang, A.-J., Lei, X.-X., Zou, K.-H. & Ng, S. W. (2004). *Acta Cryst.* E**60**, o613–o615.

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supplementary materials

Acta Cryst. (2007). E**63**, o3501 [**doi:10.1107/S1600536807033879**]

3-(4-Fluorobenzyl)-4-(2-methoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

R. A. H. Khan, R. Iqbal, Habib-ur-Rehman, G. Qadeer and W.-Y. Wong

Comment

Substituted triazole derivatives display significant biological activity including antimicrobial (Holla *et al.*, 1998), analgesic (Turan-Zitouni *et al.*, 1999), antitumor (Demirbas *et al.*, 2002), antihypertensive (Paulvannan *et al.*, 2000) and antiviral activities (Kritsanida *et al.*, 2002). The biological activity is closely related to the structure, possibly being due to the presence of the —N—C ═S unit (Omar *et al.*, 1986). We are interested in the synthesis and biological activity of aryloxyacetyl hydrazide derivatives and report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987), and are comparable with those observed in related structures (Ozturk *et al.*, 2004*a*,b).

The C8═S1 [1.6738 (17) Å] bond is in accordance with the corresponding values of 1.6773 (19) Å in 4-(4-chlorophenyl)-3-(furan-2-yl)-1*H*-1,2,4- triazole-5(4*H*)-thione (Ozturk *et al.*, 2004*a*) and 1.668 (5) Å in 4-amino-3-(1,2,3,4,5-pentahydroxypentyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (Zhang *et al.*, 2004). In the triazole ring, the N3 ═C9 [1.300 (2) Å] bond shows double-bond character.

The rings A (N1—N3/C8/C9), B (C2—C7) and C (C11—C16) are, of course, planar and dihedral angles between them are A/B = 89.96 (3)°, A/C = 74.42 (3)° and B/C = 19.58 (3)°.

In the crystal structure, intermolecular N—H···S and C—H···F hydrogen bonds (Table 1) link the molecules into infinite chains (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

The synthesis of the title compound was carried out by refluxing a solution of 1-(2-(4-fluorophenyl)acetyl)-4-(2-methoxy phenyl)thiosemicarbazide (3.33 g, 10 mmol) in 2 *M* NaOH for 5 h. Single crystals suitable for X-ray analysis were obtained by recrystallization from an aqeous ethanol solution at room temperature (yield; 78%; m.p. 464–465 K).

Refinement

H1 (for NH) was located in difference syntheses and refined isotropically [N2—H1 = 0.824 (19) Å and U_{iso}(H) = 0.069 (5) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

Figures

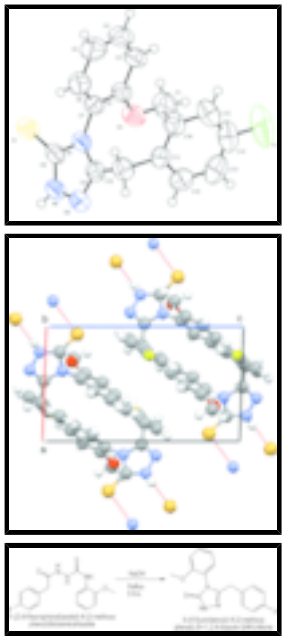


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
Fig.2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

3-(4-Fluorobenzyl)-4-(2-methoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Crystal data

$C_{16}H_{14}FN_3OS$	F_{000} = 656
M_r = 315.36	D_x = 1.340 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 464(1) K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.4910 (7) Å	Cell parameters from 5348 reflections
b = 16.1656 (15) Å	θ = 2.4–26.4º
c = 12.9147 (12) Å	μ = 0.22 mm^{-1}
β = 91.956 (2)º	T = 298 (2) K
V = 1563.0 (3) Å^3	Block, colourless
Z = 4	0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area-dedector diffractometer	2735 independent reflections
Radiation source: fine-focus sealed tube	2209 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.016
T = 298(2) K	θ_{max} = 25.0º
ω and φ scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→8

T_{min} = 0.873, T_{max} = 0.950	k = −19→9
7491 measured reflections	l = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.033	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.091	$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.323P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2735 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
203 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
S1	0.06617 (6)	0.05788 (3)	0.15775 (4)	0.07306 (18)
O1	0.30466 (16)	0.27259 (7)	0.13357 (9)	0.0683 (3)
N1	0.38497 (16)	0.11534 (7)	0.08812 (10)	0.0531 (3)
N2	0.2222 (2)	0.05914 (9)	−0.02932 (11)	0.0610 (4)
H1	0.141 (2)	0.0337 (11)	−0.0595 (15)	0.069 (5)*
N3	0.3773 (2)	0.07973 (9)	−0.07693 (11)	0.0644 (4)
F1	0.7511 (3)	0.48608 (9)	0.02635 (19)	0.1811 (9)
C1	0.2793 (3)	0.35942 (12)	0.14504 (18)	0.0876 (6)
H1A	0.2010	0.3793	0.0902	0.131*
H1B	0.3925	0.3870	0.1421	0.131*
H1C	0.2274	0.3704	0.2106	0.131*
C2	0.4123 (2)	0.23355 (10)	0.20422 (12)	0.0547 (4)
C3	0.4880 (2)	0.26997 (12)	0.29317 (13)	0.0662 (5)
H3A	0.4650	0.3252	0.3080	0.079*
C4	0.5963 (3)	0.22433 (15)	0.35849 (15)	0.0792 (6)

H4A	0.6462	0.2490	0.4178	0.095*
C5	0.6325 (3)	0.14310 (15)	0.33837 (16)	0.0866 (6)
H5A	0.7066	0.1131	0.3838	0.104*
C6	0.5588 (2)	0.10541 (12)	0.25025 (15)	0.0719 (5)
H6A	0.5821	0.0501	0.2363	0.086*
C7	0.4511 (2)	0.15124 (10)	0.18398 (12)	0.0532 (4)
C8	0.2221 (2)	0.07788 (9)	0.07140 (12)	0.0550 (4)
C9	0.4741 (2)	0.11411 (9)	−0.00335 (13)	0.0564 (4)
C10	0.6603 (2)	0.14446 (11)	−0.01445 (15)	0.0667 (5)
H10A	0.7006	0.1283	−0.0821	0.080*
H10B	0.7370	0.1172	0.0371	0.080*
C11	0.6828 (2)	0.23688 (10)	−0.00278 (13)	0.0563 (4)
C12	0.6120 (3)	0.29033 (13)	−0.07650 (15)	0.0731 (5)
H12A	0.5470	0.2694	−0.1332	0.088*
C13	0.6366 (3)	0.37436 (14)	−0.0671 (2)	0.0961 (7)
H13A	0.5907	0.4105	−0.1174	0.115*
C14	0.7288 (4)	0.40306 (13)	0.0170 (2)	0.1012 (7)
C15	0.7993 (3)	0.35367 (14)	0.0920 (2)	0.0924 (7)
H15A	0.8606	0.3758	0.1493	0.111*
C16	0.7777 (2)	0.26923 (11)	0.08116 (15)	0.0699 (5)
H16A	0.8277	0.2338	0.1310	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0624 (3)	0.0913 (4)	0.0649 (3)	−0.0217 (2)	−0.0057 (2)	−0.0104 (2)
O1	0.0728 (7)	0.0605 (7)	0.0703 (7)	0.0086 (6)	−0.0161 (6)	−0.0076 (6)
N1	0.0534 (7)	0.0482 (7)	0.0570 (8)	−0.0037 (6)	−0.0081 (6)	−0.0051 (6)
N2	0.0668 (9)	0.0547 (8)	0.0606 (9)	−0.0108 (7)	−0.0120 (7)	−0.0068 (6)
N3	0.0756 (9)	0.0557 (8)	0.0616 (8)	−0.0073 (7)	−0.0012 (7)	−0.0073 (7)
F1	0.223 (2)	0.0594 (9)	0.261 (2)	−0.0284 (10)	−0.0003 (18)	0.0010 (11)
C1	0.1005 (15)	0.0630 (12)	0.0987 (15)	0.0142 (10)	−0.0065 (12)	−0.0051 (11)
C2	0.0479 (8)	0.0622 (10)	0.0538 (9)	−0.0068 (7)	−0.0018 (7)	−0.0046 (7)
C3	0.0608 (10)	0.0775 (12)	0.0602 (10)	−0.0129 (9)	0.0007 (8)	−0.0153 (9)
C4	0.0718 (12)	0.1044 (16)	0.0603 (11)	−0.0187 (11)	−0.0144 (9)	−0.0084 (11)
C5	0.0796 (13)	0.1053 (17)	0.0727 (13)	−0.0062 (12)	−0.0295 (10)	0.0160 (12)
C6	0.0698 (11)	0.0690 (11)	0.0759 (12)	−0.0011 (9)	−0.0147 (9)	0.0092 (9)
C7	0.0483 (8)	0.0581 (9)	0.0527 (8)	−0.0081 (7)	−0.0053 (7)	−0.0016 (7)
C8	0.0573 (9)	0.0456 (8)	0.0612 (10)	−0.0029 (7)	−0.0124 (7)	−0.0031 (7)
C9	0.0632 (9)	0.0453 (8)	0.0605 (10)	0.0002 (7)	−0.0012 (8)	−0.0027 (7)
C10	0.0620 (10)	0.0619 (10)	0.0766 (12)	0.0005 (8)	0.0072 (8)	−0.0069 (9)
C11	0.0489 (8)	0.0581 (9)	0.0621 (10)	−0.0014 (7)	0.0063 (7)	0.0024 (8)
C12	0.0714 (11)	0.0802 (13)	0.0676 (11)	0.0055 (9)	0.0007 (9)	0.0058 (10)
C13	0.1059 (17)	0.0745 (15)	0.1082 (18)	0.0126 (12)	0.0100 (14)	0.0312 (13)
C14	0.1111 (18)	0.0557 (13)	0.137 (2)	−0.0129 (12)	0.0083 (17)	0.0055 (14)
C15	0.0853 (14)	0.0778 (14)	0.1133 (18)	−0.0219 (11)	−0.0074 (12)	−0.0179 (13)
C16	0.0611 (10)	0.0703 (11)	0.0776 (12)	−0.0053 (8)	−0.0070 (9)	0.0046 (9)

Geometric parameters (Å, °)

C1—O1	1.425 (2)	C9—N3	1.300 (2)
C1—H1A	0.9600	C9—N1	1.377 (2)
C1—H1B	0.9600	C9—C10	1.490 (2)
C1—H1C	0.9600	C10—C11	1.510 (2)
C2—O1	1.3529 (19)	C10—H10A	0.9700
C2—C7	1.389 (2)	C10—H10B	0.9700
C2—C3	1.394 (2)	C11—C12	1.378 (2)
C3—C4	1.366 (3)	C11—C16	1.379 (2)
C3—H3A	0.9300	C12—C13	1.376 (3)
C4—C5	1.368 (3)	C12—H12A	0.9300
C4—H4A	0.9300	C13—C14	1.349 (3)
C5—C6	1.389 (3)	C13—H13A	0.9300
C5—H5A	0.9300	C14—C15	1.349 (3)
C6—C7	1.373 (2)	C14—F1	1.357 (3)
C6—H6A	0.9300	C15—C16	1.381 (3)
C7—N1	1.4397 (19)	C15—H15A	0.9300
C8—N2	1.336 (2)	C16—H16A	0.9300
C8—N1	1.3725 (19)	N2—N3	1.374 (2)
C8—S1	1.6738 (17)	N2—H1	0.824 (19)
O1—C1—H1A	109.5	C9—C10—H10A	108.6
O1—C1—H1B	109.5	C11—C10—H10A	108.6
H1A—C1—H1B	109.5	C9—C10—H10B	108.6
O1—C1—H1C	109.5	C11—C10—H10B	108.6
H1A—C1—H1C	109.5	H10A—C10—H10B	107.6
H1B—C1—H1C	109.5	C12—C11—C16	118.76 (17)
O1—C2—C7	116.35 (13)	C12—C11—C10	120.82 (16)
O1—C2—C3	125.11 (16)	C16—C11—C10	120.41 (16)
C7—C2—C3	118.54 (15)	C13—C12—C11	120.7 (2)
C4—C3—C2	119.80 (18)	C13—C12—H12A	119.6
C4—C3—H3A	120.1	C11—C12—H12A	119.6
C2—C3—H3A	120.1	C14—C13—C12	118.3 (2)
C3—C4—C5	121.25 (17)	C14—C13—H13A	120.9
C3—C4—H4A	119.4	C12—C13—H13A	120.9
C5—C4—H4A	119.4	C15—C14—C13	123.5 (2)
C4—C5—C6	120.07 (18)	C15—C14—F1	118.5 (3)
C4—C5—H5A	120.0	C13—C14—F1	118.0 (3)
C6—C5—H5A	120.0	C14—C15—C16	118.1 (2)
C7—C6—C5	118.86 (18)	C14—C15—H15A	120.9
C7—C6—H6A	120.6	C16—C15—H15A	120.9
C5—C6—H6A	120.6	C11—C16—C15	120.64 (19)
C6—C7—C2	121.47 (15)	C11—C16—H16A	119.7
C6—C7—N1	119.73 (15)	C15—C16—H16A	119.7
C2—C7—N1	118.69 (13)	C8—N1—C9	108.26 (13)
N2—C8—N1	102.91 (14)	C8—N1—C7	126.22 (13)
N2—C8—S1	128.94 (12)	C9—N1—C7	125.52 (13)
N1—C8—S1	128.12 (12)	C8—N2—N3	114.16 (14)

N3—C9—N1	111.00 (14)	C8—N2—H1	123.4 (13)
N3—C9—C10	124.82 (16)	N3—N2—H1	122.3 (13)
N1—C9—C10	124.11 (14)	C9—N3—N2	103.63 (14)
C9—C10—C11	114.68 (14)	C2—O1—C1	117.90 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···S1^{i}	0.824 (19)	2.465 (17)	3.2789 (16)	171.1 (16)
C5—H5A···F1^{ii}	0.93	2.37	3.186 (3)	146

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.

Fig. 1

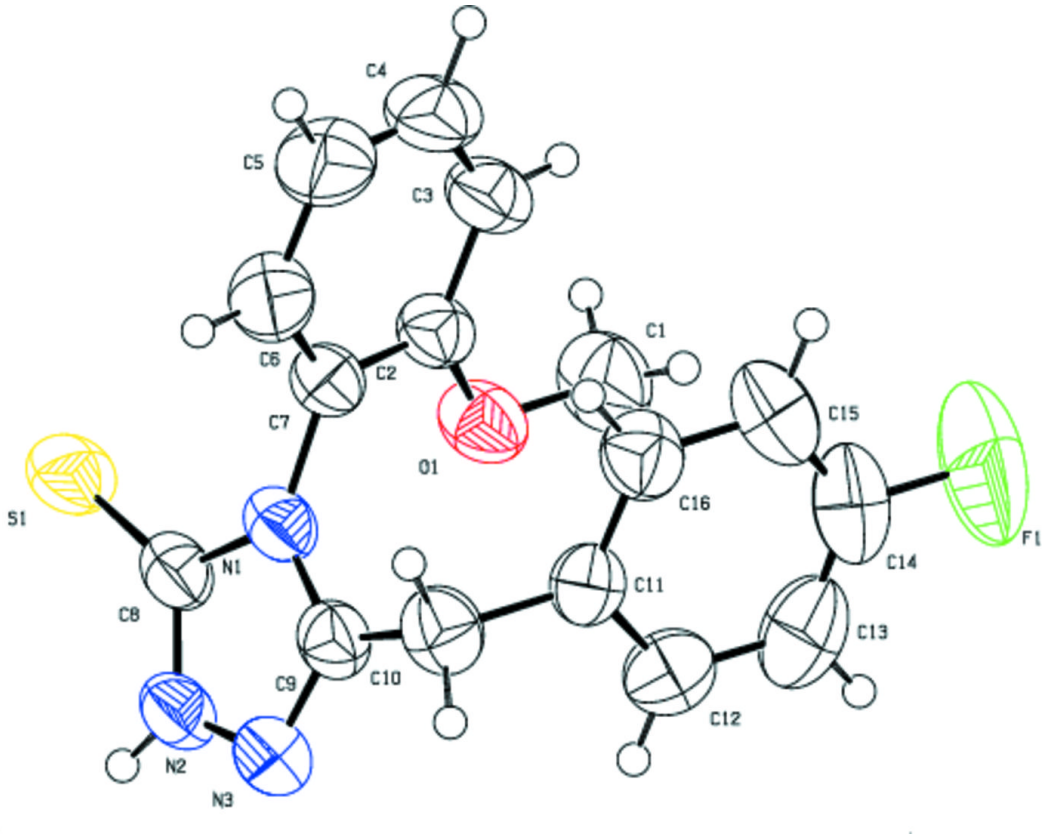

C4
C5
C3
C6
C2
C1
C7
O1
C15
S1
C16
F1
N1
C14
C11
C8
C13
C9
C10
C12
N2
N3

Fig. 2

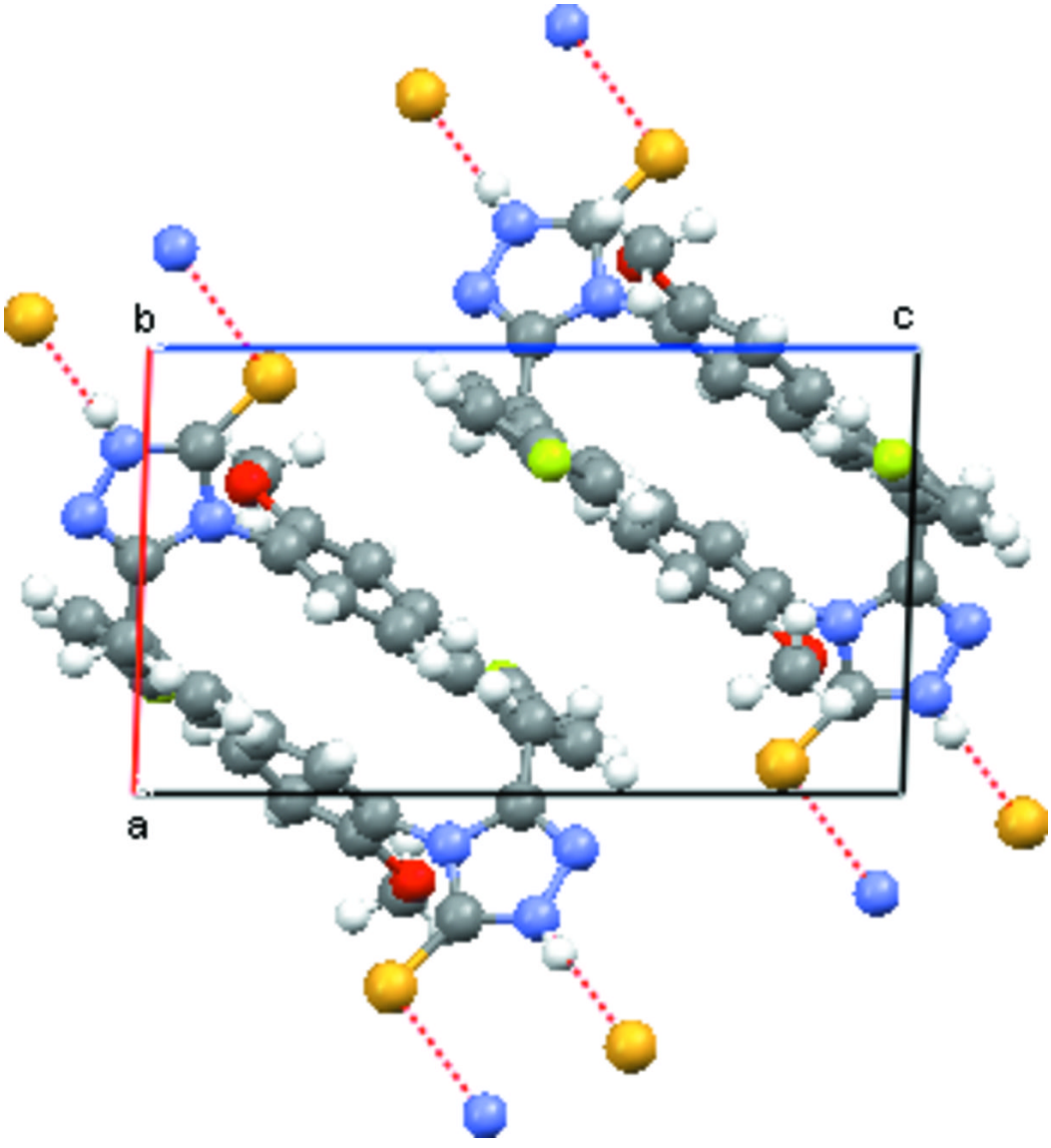
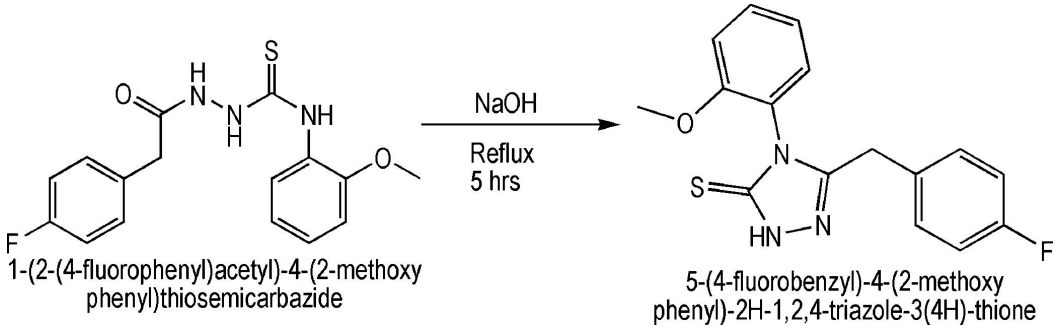


Fig. 3



1-(2-(4-fluorophenyl)acetyl)-4-(2-methoxy phenyl)thiosemicarbazide

5-(4-fluorobenzyl)-4-(2-methoxy phenyl)-2H-1,2,4-triazole-3(4H)-thione