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4-(4-Chlorophenyl)-3-(3-methoxyphenethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

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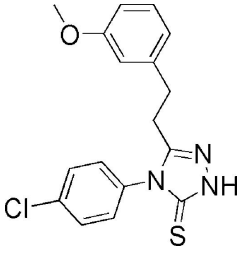
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.111; data-to-parameter ratio = 18.0.

The title compound, $C_{17}H_{16}ClN_3OS$, is an important biologically active heterocyclic compound. The triazole ring is oriented with respect to the 4-chlorophenyl and 2-(3-methoxyphenyl)ethyl rings at dihedral angles of 89.5 (1) and 56.9 (1)°, respectively. The dihedral angle between the benzene rings is 78.5 (1)°. In the crystal structure, intermolecular N—H···S hydrogen bonds link the molecules.

Related literature

For related literature, 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); Öztürk *et al.* (2004*a*,*b*); Zhang *et al.* (2004).



Experimental

Crystal data

$C_{17}H_{16}ClN_3OS$
M_r = 345.84
Triclinic, $P\overline{1}$
a = 7.1697 (3) Å
b = 11.1376 (4) Å
c = 11.7574 (5) Å
α = 67.070 (2)°
β = 77.421 (2)°
γ = 72.948 (3)°
V = 820.93 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 120 (2) K
0.26 × 0.18 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.912, T_{max} = 0.953
15238 measured reflections
3752 independent reflections
2725 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.04
3752 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
S1—H1S···N2^{i}	1.00	2.37	3.2546 (18)	147

Symmetry code: (i) $-x+3, -y-1, -z+2$.

Data collection: *COLLECT* (Bruker, 2000); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2007); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2353).

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supplementary materials

Acta Cryst. (2007). E**63**, o4507 [**doi:10.1107/S1600536807053329**]

4-(4-Chlorophenyl)-3-(3-methoxyphenethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

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Comment

Substituted triazole derivatives display significant biological activities 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 activities of aryloxyacetyl hydrazide derivatives and report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of the title compound, (I), (Fig. 1) the bond lengths and angles are comparable with those observed in related structures (Öztürk *et al.*, 2004*a*,b). The C1═S1 [1.680 (2) Å] bond agrees with the corresponding values [1.6773 (19) Å] in 4-(4-chlorophenyl)-3-(furan-2-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione (Öztürk *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 N2 ═C2 [1.347 (3) Å] bond has double-bond character. The planar triazole ring A (N1–N3/C7/C8) is oriented with respect to planar 4-chlorophenyl and 2-(3-methoxyphenyl)ethyl rings; B (C1–C6) and C (C11–C16), at dihedral angles of 89.5 (1) and 56.9 (1)°, respectively. The dihedral angle between rings B and C is B/C = 78.5 (1)°.

In the crystal structure, intermolecular N—H···S hydrogen bonds (Table 1, Fig. 2) link the molecules, in which they seem to be effective in the stabilization of the structure.

Experimental

The synthesis of the title compound was carried out by refluxing a solution of 1-(3-(3-methoxyphenyl)propanoyl)-4-(4-chlorophenyl)thiosemicarbazide (3.63 g, 10 mmol) in NaOH (2 *M*) for 5 h. Single crystals suitable for X-ray analysis were obtained by recrystallization from an aqeous ethanol solution at room temperature (yield; 72%, m.p. 461–462 K).

Refinement

The H atom (for SH) was located in difference syntheses, and constrained to ride on its parent atom, with S—H = 0.9972 Å and U_{iso} = 1.5U_{eq}(S). The remaining H atoms were positioned geometrically, with C—H = 0.95, 0.99 and 0.98 Å for aromatic, methylene and methyl H atoms, respectively, 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

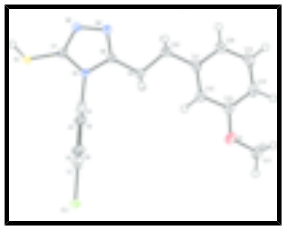


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

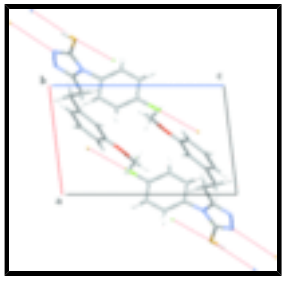


Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Fig. 3. The formation of the title compound.

4-(4-Chlorophenyl)-3-(3-methoxyphenethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Crystal data

$C_{17}H_{16}ClN_3OS$	$Z = 2$
$M_r = 345.84$	$F_{000} = 360$
Triclinic, $P\overline{1}$	$D_x = 1.399$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 461(1) K
$a = 7.1697$ (3) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.1376$ (4) Å	Cell parameters from 9213 reflections
$c = 11.7574$ (5) Å	$\theta = 1.0$–27.5º
$\alpha = 67.070$ (2)º	$\mu = 0.37$ mm^{-1}
$\beta = 77.421$ (2)º	$T = 120$ (2) K
$\gamma = 72.948$ (3)º	Block, colorless
$V = 820.93$ (6) Å^3	0.26 × 0.18 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer	3752 independent reflections
Radiation source: fine-focus sealed tube	2725 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.048$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 120(2)$ K	$\theta_{min} = 1.9$º
φ scans and ω scans with κ offset	$h = -9 \rightarrow 9$

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.912, T_{max} = 0.953

15238 measured reflections

$k = -14 \rightarrow 14$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.043

$wR(F^2)$ = 0.111

S = 1.04

3752 reflections

209 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.5947P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.74 e Å^{-3}

$\Delta\rho_{min}$ = −0.77 e Å^{-3}

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 $F^2 > 2$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}
Cl1	0.76936 (8)	0.01723 (5)	0.38434 (5)	0.02439 (16)
S1	1.42541 (8)	−0.32374 (5)	0.81974 (5)	0.02415 (16)
H1S	1.4839	−0.3213	0.8882	0.036*
O1	0.3991 (2)	−0.62525 (16)	0.62402 (16)	0.0305 (4)
N1	1.1468 (2)	−0.43495 (17)	0.79786 (15)	0.0162 (4)
N2	1.3112 (3)	−0.55857 (17)	0.95049 (16)	0.0186 (4)
N3	1.1823 (3)	−0.63067 (17)	0.95225 (16)	0.0192 (4)
C1	0.8828 (3)	−0.1146 (2)	0.5053 (2)	0.0182 (5)
C2	1.0559 (3)	−0.1999 (2)	0.4786 (2)	0.0201 (5)
H2	1.1130	−0.1859	0.3949	0.024*
C3	1.1453 (3)	−0.3063 (2)	0.5751 (2)	0.0193 (5)
H3	1.2630	−0.3667	0.5583	0.023*
C4	1.0604 (3)	−0.3228 (2)	0.69584 (19)	0.0162 (4)
C5	0.8906 (3)	−0.2357 (2)	0.7231 (2)	0.0203 (5)
H5	0.8371	−0.2475	0.8071	0.024*

C6	0.7986 (3)	−0.1308 (2)	0.6265 (2)	0.0211 (5)
H6	0.6800	−0.0710	0.6434	0.025*
C7	1.2942 (3)	−0.4394 (2)	0.85785 (19)	0.0180 (5)
C8	1.0843 (3)	−0.5532 (2)	0.85786 (19)	0.0175 (4)
C9	0.9291 (3)	−0.5843 (2)	0.8159 (2)	0.0203 (5)
H9A	0.9608	−0.5699	0.7260	0.024*
H9B	0.8021	−0.5219	0.8278	0.024*
C10	0.9083 (3)	−0.7281 (2)	0.8865 (2)	0.0226 (5)
H10A	1.0385	−0.7895	0.8802	0.027*
H10B	0.8676	−0.7398	0.9755	0.027*
C11	0.7628 (3)	−0.7685 (2)	0.8412 (2)	0.0209 (5)
C12	0.6384 (3)	−0.6796 (2)	0.7539 (2)	0.0211 (5)
H12	0.6405	−0.5874	0.7214	0.025*
C13	0.5102 (3)	−0.7227 (2)	0.7129 (2)	0.0224 (5)
C14	0.5016 (3)	−0.8572 (2)	0.7609 (2)	0.0258 (5)
H14	0.4141	−0.8872	0.7336	0.031*
C15	0.6245 (4)	−0.9462 (2)	0.8498 (2)	0.0301 (6)
H15	0.6197	−1.0380	0.8840	0.036*
C16	0.7531 (4)	−0.9040 (2)	0.8892 (2)	0.0283 (5)
H16	0.8363	−0.9671	0.9495	0.034*
C17	0.2965 (4)	−0.6684 (3)	0.5611 (3)	0.0376 (6)
H17A	0.3901	−0.7301	0.5230	0.056*
H17B	0.2303	−0.5905	0.4963	0.056*
H17C	0.1988	−0.7143	0.6208	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0265 (3)	0.0187 (3)	0.0252 (3)	−0.0051 (2)	−0.0144 (2)	0.0011 (2)
S1	0.0245 (3)	0.0206 (3)	0.0261 (3)	−0.0096 (2)	−0.0142 (2)	0.0023 (2)
O1	0.0293 (9)	0.0269 (9)	0.0428 (10)	−0.0037 (7)	−0.0177 (8)	−0.0150 (8)
N1	0.0168 (9)	0.0155 (9)	0.0157 (9)	−0.0046 (7)	−0.0057 (7)	−0.0021 (7)
N2	0.0191 (9)	0.0168 (9)	0.0203 (9)	−0.0069 (7)	−0.0059 (7)	−0.0029 (7)
N3	0.0207 (9)	0.0184 (9)	0.0190 (9)	−0.0060 (7)	−0.0052 (7)	−0.0045 (7)
C1	0.0217 (11)	0.0132 (10)	0.0212 (11)	−0.0070 (8)	−0.0105 (9)	−0.0011 (8)
C2	0.0223 (11)	0.0201 (11)	0.0182 (11)	−0.0045 (9)	−0.0053 (9)	−0.0059 (9)
C3	0.0181 (11)	0.0185 (11)	0.0210 (11)	−0.0029 (8)	−0.0049 (9)	−0.0062 (9)
C4	0.0162 (10)	0.0143 (10)	0.0187 (10)	−0.0057 (8)	−0.0076 (8)	−0.0019 (8)
C5	0.0198 (11)	0.0204 (11)	0.0186 (11)	−0.0054 (9)	−0.0019 (9)	−0.0043 (9)
C6	0.0165 (11)	0.0199 (11)	0.0255 (12)	−0.0035 (8)	−0.0036 (9)	−0.0062 (9)
C7	0.0157 (10)	0.0190 (11)	0.0175 (10)	−0.0021 (8)	−0.0041 (8)	−0.0046 (9)
C8	0.0186 (11)	0.0162 (10)	0.0164 (10)	−0.0054 (8)	−0.0025 (8)	−0.0032 (8)
C9	0.0214 (11)	0.0198 (11)	0.0211 (11)	−0.0071 (9)	−0.0074 (9)	−0.0044 (9)
C10	0.0265 (12)	0.0213 (11)	0.0211 (11)	−0.0086 (9)	−0.0065 (9)	−0.0045 (9)
C11	0.0219 (11)	0.0212 (11)	0.0212 (11)	−0.0087 (9)	0.0012 (9)	−0.0084 (9)
C12	0.0226 (11)	0.0198 (11)	0.0237 (11)	−0.0073 (9)	−0.0023 (9)	−0.0091 (9)
C13	0.0189 (11)	0.0255 (12)	0.0250 (12)	−0.0040 (9)	−0.0010 (9)	−0.0128 (10)
C14	0.0257 (12)	0.0286 (13)	0.0310 (13)	−0.0120 (10)	−0.0002 (10)	−0.0162 (10)

C15	0.0362 (14)	0.0234 (12)	0.0333 (14)	−0.0146 (11)	−0.0014 (11)	−0.0084 (11)
C16	0.0331 (13)	0.0231 (12)	0.0281 (13)	−0.0109 (10)	−0.0077 (11)	−0.0030 (10)
C17	0.0365 (15)	0.0410 (15)	0.0469 (16)	−0.0090 (12)	−0.0200 (13)	−0.0197 (13)

Geometric parameters (Å, °)

Cl1—C1	1.737 (2)	C8—C9	1.483 (3)
S1—C7	1.680 (2)	C9—C10	1.525 (3)
S1—H1S	0.9972	C9—H9A	0.9900
O1—C13	1.370 (3)	C9—H9B	0.9900
O1—C17	1.429 (3)	C10—C11	1.509 (3)
N1—C7	1.372 (3)	C10—H10A	0.9900
N1—C8	1.385 (3)	C10—H10B	0.9900
N1—C4	1.444 (2)	C11—C12	1.384 (3)
N2—C7	1.341 (3)	C11—C16	1.408 (3)
N2—N3	1.384 (2)	C12—C13	1.393 (3)
N3—C8	1.307 (3)	C12—H12	0.9500
C1—C2	1.383 (3)	C13—C14	1.396 (3)
C1—C6	1.384 (3)	C14—C15	1.390 (3)
C2—C3	1.387 (3)	C14—H14	0.9500
C2—H2	0.9500	C15—C16	1.377 (3)
C3—C4	1.380 (3)	C15—H15	0.9500
C3—H3	0.9500	C16—H16	0.9500
C4—C5	1.381 (3)	C17—H17A	0.9800
C5—C6	1.390 (3)	C17—H17B	0.9800
C5—H5	0.9500	C17—H17C	0.9800
C6—H6	0.9500		
C7—S1—H1S	116.9	C8—C9—H9B	109.1
C13—O1—C17	117.19 (18)	C10—C9—H9B	109.1
C7—N1—C8	108.24 (16)	H9A—C9—H9B	107.9
C7—N1—C4	126.25 (17)	C11—C10—C9	114.60 (18)
C8—N1—C4	125.36 (17)	C11—C10—H10A	108.6
C7—N2—N3	113.38 (17)	C9—C10—H10A	108.6
C8—N3—N2	104.10 (16)	C11—C10—H10B	108.6
C2—C1—C6	121.58 (19)	C9—C10—H10B	108.6
C2—C1—Cl1	119.36 (17)	H10A—C10—H10B	107.6
C6—C1—Cl1	119.05 (17)	C12—C11—C16	117.8 (2)
C1—C2—C3	119.4 (2)	C12—C11—C10	123.64 (19)
C1—C2—H2	120.3	C16—C11—C10	118.5 (2)
C3—C2—H2	120.3	C11—C12—C13	121.4 (2)
C4—C3—C2	119.0 (2)	C11—C12—H12	119.3
C4—C3—H3	120.5	C13—C12—H12	119.3
C2—C3—H3	120.5	O1—C13—C12	115.4 (2)
C3—C4—C5	121.81 (19)	O1—C13—C14	124.1 (2)
C3—C4—N1	120.17 (18)	C12—C13—C14	120.5 (2)
C5—C4—N1	118.01 (19)	C15—C14—C13	118.2 (2)
C4—C5—C6	119.3 (2)	C15—C14—H14	120.9
C4—C5—H5	120.3	C13—C14—H14	120.9
C6—C5—H5	120.3	C16—C15—C14	121.3 (2)

C1—C6—C5	118.9 (2)	C16—C15—H15	119.3
C1—C6—H6	120.6	C14—C15—H15	119.3
C5—C6—H6	120.6	C15—C16—C11	120.8 (2)
N2—C7—N1	103.63 (18)	C15—C16—H16	119.6
N2—C7—S1	128.85 (16)	C11—C16—H16	119.6
N1—C7—S1	127.50 (15)	O1—C17—H17A	109.5
N3—C8—N1	110.66 (18)	O1—C17—H17B	109.5
N3—C8—C9	126.37 (18)	H17A—C17—H17B	109.5
N1—C8—C9	122.96 (18)	O1—C17—H17C	109.5
C8—C9—C10	112.31 (17)	H17A—C17—H17C	109.5
C8—C9—H9A	109.1	H17B—C17—H17C	109.5
C10—C9—H9A	109.1		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
S1—H1S···N2^{i}	1.00	2.37	3.2546 (18)	147

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

Fig. 1

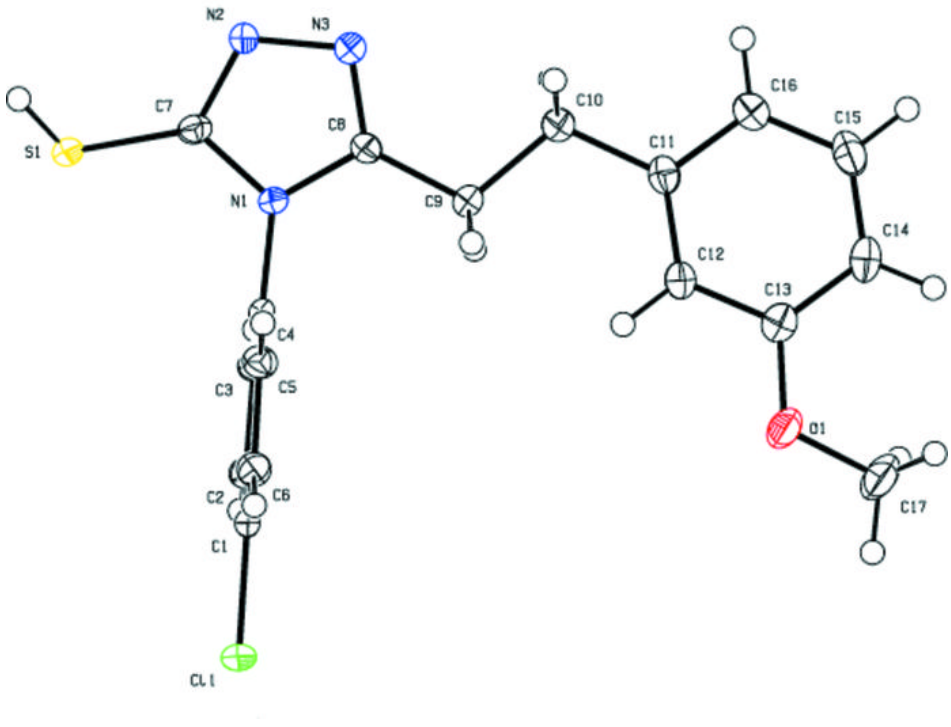

N2
N3
C7
C8
C10
C16
S1
C11
C15
N1
C9
C14
C12
C13
C4
C3
C5
O1
C2
C6
C17
C1
Cl1

Fig. 2

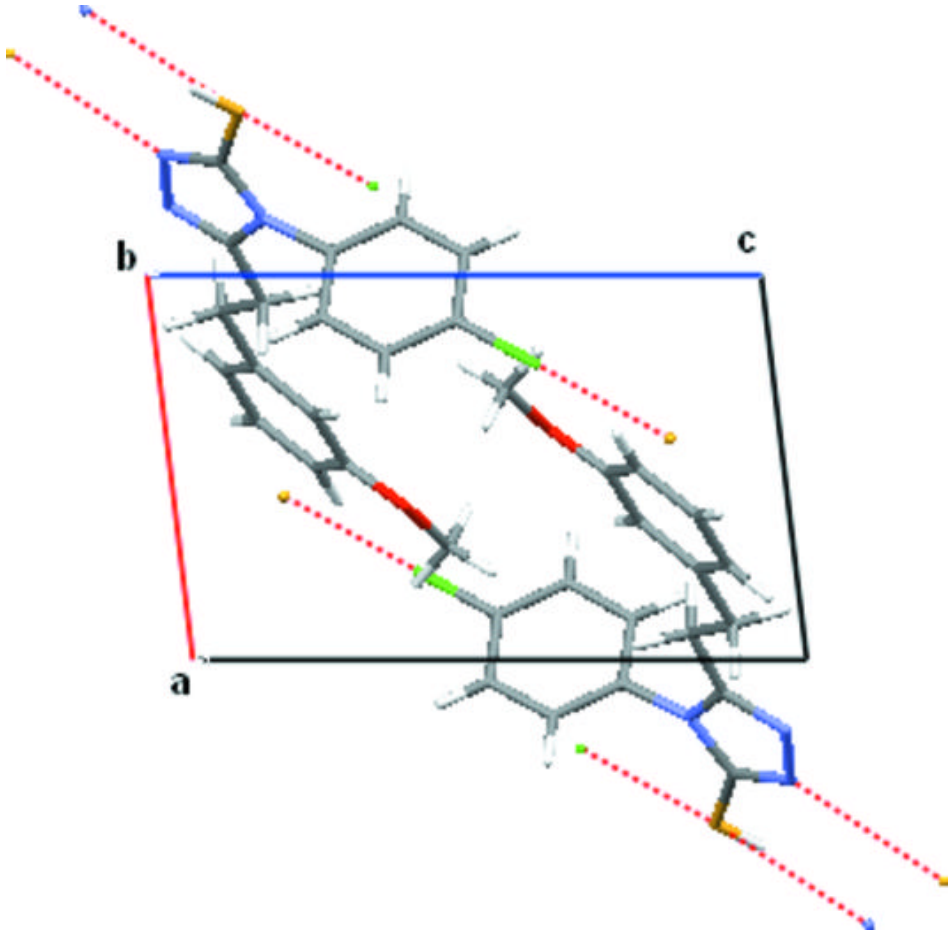
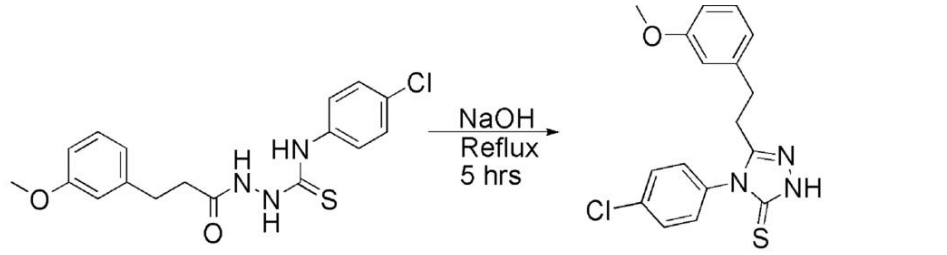


Fig. 3



1-(3-(3-methoxyphenyl)propanoyl)-4-(4-chlorophenyl)thiosemicarbazide

5-(3-methoxyphenethyl)-4-(4-chlorophenyl)-2*H*-1,2,4-triazole-3(4*H*)-thione