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

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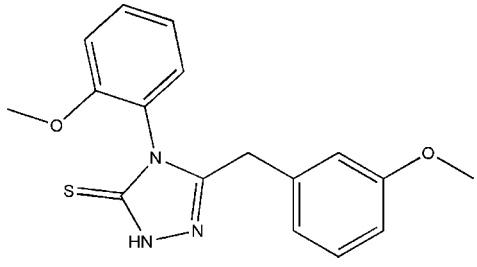
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.073; wR factor = 0.159; data-to-parameter ratio = 17.8.

In the title compound, $C_{17}H_{17}N_3O_2S$, the five-membered ring forms dihedral angles of 53.02 (3) and 78.57 (3)° with the 3-methoxy-substituted and 2-methoxy-substituted benzene rings, respectively. In the crystal structure, molecules are linked into centrosymmetric dimers *via* intermolecular N—H···S hydrogen bonds.

Related literature

For background information on the biological activity of substituted triazole derivatives, see: Demirbas *et al.* (2002); Holla *et al.* (1998); Omar *et al.* (1986); Paulvannan *et al.* (2000); Turan-Zitouni *et al.* (1999); Kritsanida *et al.* (2002). For related structures, see: Öztürk *et al.* (2004*a*,*b*); Zhang *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{17}H_{17}N_3O_2S$
M_r = 327.40
Triclinic, $P\overline{1}$
a = 7.3941 (3) Å
b = 10.6459 (5) Å
c = 12.1940 (8) Å
α = 68.841 (5)°
β = 74.317 (5)°
γ = 75.187 (5)°
V = 848.37 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 293 (2) K
0.40 × 0.24 × 0.15 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: integration (Gaussian; Coppens, 1970)
T_{min} = 0.946, T_{max} = 0.983
10764 measured reflections
3708 independent reflections
2064 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.159
S = 1.10
3708 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.86	2.42	3.277 (3)	172

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

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); 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: LH2729).

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supplementary materials

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

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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 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 substituted triazole derivatives and report here the synthesis and crystal structure of the title compound, (I) (Fig. 1).

In the molecluar structure of (I), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and comparable with those observed in related structures (Öztürk *et al.*, 2004*a*,b). The C7-S1 bond length [1.679 (3) A °] compares with 1.6773 (19) A ° 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) A ° 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-C8 bond [1.294 (4)] bond shows the expected double bond character.

The rings A (N1—N3/C7/C8), B (C1—C6) and C (C10—C15) are eesentially planar and dihedral angles between them are A/B = 78.57 (3)°, A/C = 53.02 (3)° and B/C = 16.23 (3)°. In the crystal structure, molecules are linked into centrosymmetric dimers via intermolecular N–H···S hydrogen bonds.

Experimental

The synthesis of the title compound was carried out by refluxing a solution of 4-(2-methoxyphenyl)-1-(2-(3-methoxyphenyl)acetyl)thiosemicarbazide (3.45 g, 10 mmol) in 2 *M* NaOH for 5 h. Single crystals suitable for X-ray measurements were obtained by recrystallization from an aqeous ethanol solution at room temperature (yield: 75%; m.p. 469–470 K).

Refinement

H atoms were placed in calculated positions with C-H = 0.93-0.97Å and N-H = 0.86Å and included in the refinement with $U_{iso}(H) = 1.2U_{eq}(C,N)$. Although the atoms of substituted methoxy have larger than normal anisotropic displacement parameters, attempts to create disorder models did not improve the precision of the structure.

Figures

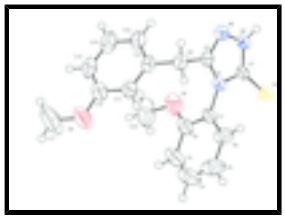


Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

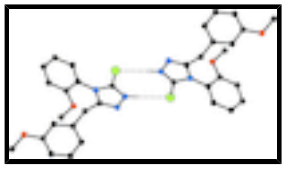


Fig. 2. Part of the crystal structure of (I) showing hydrogen bonds as dashed lines.

Fig. 3. The reaction scheme.

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

Crystal data

$C_{17}H_{17}N_3O_2S$	$Z = 2$
$M_r = 327.40$	$F(000) = 344$
Triclinic, $P\overline{1}$	$D_x = 1.282$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 469(1) K
$a = 7.3941$ (3) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.6459$ (5) Å	Cell parameters from 10808 reflections
$c = 12.1940$ (8) Å	$\theta = 1$–$27.5°$
$\alpha = 68.841$ (5)°	$\mu = 0.20$ mm^{-1}
$\beta = 74.317$ (5)°	$T = 293$ K
$\gamma = 75.187$ (5)°	Plate, colourless
$V = 848.37$ (8) Å^3	0.40 × 0.24 × 0.15 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	3708 independent reflections
Radiation source: fine-focus sealed tube	2064 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.079$
Detector resolution: 9.091 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
φ and ω scans to fill the Ewald sphere	$h = -9 \rightarrow 9$
Absorption correction: integration (Gaussian; Coppens, 1970)	$k = -13 \rightarrow 13$
$T_{min} = 0.946$, $T_{max} = 0.983$	$l = -15 \rightarrow 15$
10764 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.073$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.159$	H-atom parameters constrained
$S = 1.10$	$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.6393P]$ where $P = (F_o^2 + 2F_c^2)/3$
3708 reflections	$(\Delta/\sigma)_{max} < 0.001$
208 parameters	$\Delta\rho_{max} = 0.32$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.24$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	−0.37112 (12)	0.55979 (11)	0.30006 (8)	0.0515 (3)
N2	−0.2330 (4)	0.4093 (3)	0.5026 (2)	0.0410 (7)
H2	−0.3425	0.4150	0.5499	0.049*
N1	−0.0166 (3)	0.4302 (3)	0.3462 (2)	0.0379 (6)
N3	−0.0666 (4)	0.3402 (3)	0.5425 (2)	0.0444 (7)
C7	−0.2087 (4)	0.4659 (3)	0.3847 (3)	0.0362 (7)
C10	0.3280 (4)	0.1679 (3)	0.4006 (3)	0.0416 (8)
C1	0.0831 (4)	0.4687 (4)	0.2239 (3)	0.0460 (9)
C8	0.0625 (4)	0.3535 (3)	0.4452 (3)	0.0374 (7)
C11	0.4677 (5)	0.1629 (4)	0.3017 (3)	0.0535 (9)
H11	0.5259	0.2385	0.2566	0.064*
O1	0.0235 (4)	0.2702 (3)	0.2152 (3)	0.0745 (8)
C14	0.2995 (6)	−0.0616 (4)	0.4330 (4)	0.0700 (12)
H14	0.2433	−0.1380	0.4789	0.084*
C9	0.2686 (4)	0.2945 (3)	0.4391 (3)	0.0446 (8)
H9A	0.2988	0.2723	0.5173	0.054*
H9B	0.3424	0.3630	0.3829	0.054*
C15	0.2434 (5)	0.0546 (4)	0.4662 (4)	0.0591 (11)

H15	0.1477	0.0574	0.5333	0.071*
C2	0.1052 (5)	0.3814 (5)	0.1581 (3)	0.0543 (10)
C3	0.2031 (6)	0.4167 (6)	0.0396 (4)	0.0767 (15)
H3	0.2209	0.3601	−0.0067	0.092*
C13	0.4385 (6)	−0.0674 (4)	0.3336 (4)	0.0634 (11)
H13	0.4743	−0.1460	0.3107	0.076*
C6	0.1539 (5)	0.5894 (4)	0.1775 (3)	0.0605 (11)
H6	0.1374	0.6459	0.2237	0.073*
C12	0.5230 (6)	0.0434 (4)	0.2686 (4)	0.0639 (11)
C5	0.2519 (6)	0.6227 (6)	0.0573 (4)	0.0808 (15)
H5	0.3011	0.7034	0.0217	0.097*
O2	0.6618 (5)	0.0512 (4)	0.1674 (3)	0.1103 (13)
C16	0.0674 (8)	0.1673 (6)	0.1580 (5)	0.106 (2)
H16A	0.0231	0.2046	0.0837	0.127*
H16B	0.0055	0.0915	0.2094	0.127*
H16C	0.2029	0.1366	0.1426	0.127*
C4	0.2727 (6)	0.5350 (7)	−0.0064 (4)	0.0838 (17)
H4	0.3387	0.5582	−0.0855	0.101*
C17	0.7351 (12)	−0.0684 (7)	0.1341 (7)	0.181 (4)
H17A	0.7803	−0.1401	0.2007	0.217*
H17B	0.8390	−0.0520	0.0666	0.217*
H17C	0.6375	−0.0952	0.1134	0.217*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0403 (5)	0.0619 (7)	0.0415 (5)	0.0027 (4)	−0.0018 (4)	−0.0162 (4)
N2	0.0386 (15)	0.0399 (17)	0.0390 (16)	−0.0030 (12)	0.0016 (12)	−0.0155 (13)
N1	0.0363 (14)	0.0392 (17)	0.0346 (15)	−0.0026 (12)	0.0005 (11)	−0.0154 (13)
N3	0.0486 (16)	0.0385 (17)	0.0425 (17)	−0.0052 (13)	−0.0046 (13)	−0.0135 (14)
C7	0.0415 (17)	0.0327 (19)	0.0339 (17)	−0.0070 (14)	0.0042 (13)	−0.0180 (15)
C10	0.0378 (17)	0.035 (2)	0.052 (2)	0.0009 (14)	−0.0134 (15)	−0.0141 (16)
C1	0.0340 (17)	0.059 (2)	0.0374 (18)	0.0044 (16)	−0.0034 (14)	−0.0182 (18)
C8	0.0442 (18)	0.0298 (18)	0.0399 (19)	−0.0055 (14)	−0.0060 (15)	−0.0152 (15)
C11	0.063 (2)	0.039 (2)	0.054 (2)	−0.0098 (17)	−0.0022 (18)	−0.0157 (18)
O1	0.086 (2)	0.076 (2)	0.076 (2)	−0.0040 (17)	−0.0174 (16)	−0.0474 (18)
C14	0.062 (2)	0.041 (2)	0.099 (3)	−0.0095 (19)	−0.010 (2)	−0.017 (2)
C9	0.0454 (19)	0.039 (2)	0.050 (2)	−0.0018 (15)	−0.0128 (16)	−0.0164 (17)
C15	0.046 (2)	0.044 (2)	0.077 (3)	−0.0036 (18)	0.0007 (19)	−0.020 (2)
C2	0.040 (2)	0.075 (3)	0.047 (2)	0.0079 (19)	−0.0101 (16)	−0.030 (2)
C3	0.049 (2)	0.124 (5)	0.050 (3)	0.015 (3)	−0.007 (2)	−0.041 (3)
C13	0.072 (3)	0.040 (2)	0.082 (3)	0.003 (2)	−0.022 (2)	−0.029 (2)
C6	0.041 (2)	0.068 (3)	0.053 (2)	−0.0121 (19)	−0.0062 (17)	0.003 (2)
C12	0.066 (3)	0.057 (3)	0.062 (3)	−0.002 (2)	0.000 (2)	−0.027 (2)
C5	0.052 (2)	0.102 (4)	0.061 (3)	−0.021 (2)	−0.005 (2)	0.007 (3)
O2	0.142 (3)	0.076 (2)	0.094 (3)	−0.024 (2)	0.042 (2)	−0.049 (2)
C16	0.129 (5)	0.094 (4)	0.130 (5)	0.021 (3)	−0.062 (4)	−0.077 (4)
C4	0.047 (2)	0.137 (5)	0.047 (3)	−0.002 (3)	0.000 (2)	−0.020 (3)

C17	0.222 (8)	0.116 (6)	0.175 (7)	−0.045 (5)	0.100 (6)	−0.102 (6)

Geometric parameters (Å, °)

S1—C7	1.679 (3)	C9—H9A	0.9700
N2—C7	1.324 (4)	C9—H9B	0.9700
N2—N3	1.377 (4)	C15—H15	0.9300
N2—H2	0.8600	C2—C3	1.390 (6)
N1—C7	1.369 (4)	C3—C4	1.353 (7)
N1—C8	1.375 (4)	C3—H3	0.9300
N1—C1	1.434 (4)	C13—C12	1.358 (6)
N3—C8	1.294 (4)	C13—H13	0.9300
C10—C11	1.367 (5)	C6—C5	1.407 (6)
C10—C15	1.380 (5)	C6—H6	0.9300
C10—C9	1.507 (5)	C12—O2	1.366 (5)
C1—C6	1.380 (5)	C5—C4	1.372 (7)
C1—C2	1.387 (5)	C5—H5	0.9300
C8—C9	1.485 (4)	O2—C17	1.407 (6)
C11—C12	1.402 (5)	C16—H16A	0.9599
C11—H11	0.9300	C16—H16B	0.9601
O1—C2	1.340 (5)	C16—H16C	0.9600
O1—C16	1.428 (5)	C4—H4	0.9300
C14—C15	1.370 (5)	C17—H17A	0.9600
C14—C13	1.370 (6)	C17—H17B	0.9600
C14—H14	0.9299	C17—H17C	0.9599
C7—N2—N3	113.7 (3)	O1—C2—C1	115.7 (3)
C7—N2—H2	123.2	O1—C2—C3	125.5 (4)
N3—N2—H2	123.1	C1—C2—C3	118.7 (5)
C7—N1—C8	108.1 (2)	C4—C3—C2	118.4 (5)
C7—N1—C1	125.4 (3)	C4—C3—H3	120.8
C8—N1—C1	126.5 (2)	C2—C3—H3	120.8
C8—N3—N2	103.9 (3)	C12—C13—C14	119.2 (4)
N2—C7—N1	103.5 (3)	C12—C13—H13	120.3
N2—C7—S1	129.2 (2)	C14—C13—H13	120.5
N1—C7—S1	127.3 (2)	C1—C6—C5	116.9 (5)
C11—C10—C15	119.2 (3)	C1—C6—H6	121.3
C11—C10—C9	120.7 (3)	C5—C6—H6	121.7
C15—C10—C9	120.1 (3)	C13—C12—O2	124.9 (4)
C6—C1—C2	123.1 (4)	C13—C12—C11	120.6 (4)
C6—C1—N1	118.8 (3)	O2—C12—C11	114.5 (4)
C2—C1—N1	118.1 (4)	C4—C5—C6	119.1 (5)
N3—C8—N1	110.8 (3)	C4—C5—H5	120.7
N3—C8—C9	125.4 (3)	C6—C5—H5	120.1
N1—C8—C9	123.8 (3)	C12—O2—C17	117.6 (4)
C10—C11—C12	119.8 (4)	O1—C16—H16A	109.5
C10—C11—H11	120.1	O1—C16—H16B	109.4
C12—C11—H11	120.1	H16A—C16—H16B	109.5
C2—O1—C16	117.5 (4)	O1—C16—H16C	109.5
C15—C14—C13	120.9 (4)	H16A—C16—H16C	109.5

C15—C14—H14	119.6	H16B—C16—H16C	109.5
C13—C14—H14	119.5	C3—C4—C5	123.7 (5)
C8—C9—C10	113.6 (3)	C3—C4—H4	118.3
C8—C9—H9A	108.9	C5—C4—H4	118.1
C10—C9—H9A	108.9	O2—C17—H17A	108.6
C8—C9—H9B	108.8	O2—C17—H17B	109.5
C10—C9—H9B	108.7	H17A—C17—H17B	109.5
H9A—C9—H9B	107.7	O2—C17—H17C	110.4
C14—C15—C10	120.4 (4)	H17A—C17—H17C	109.5
C14—C15—H15	119.9	H17B—C17—H17C	109.5
C10—C15—H15	119.7		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.86	2.42	3.277 (3)	172.

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

Fig. 1

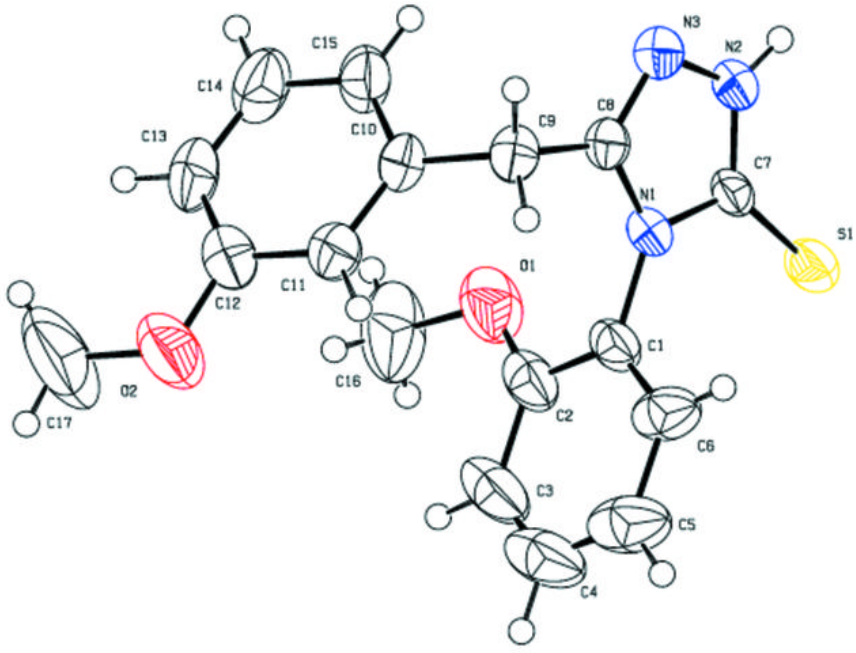

N3
N2
C15
C14
C10
C9
C8
C13
C7
N1
S1
O1
C12
C11
C1
O2
C16
C2
C6
C17
C3
C5
C4

Fig. 2

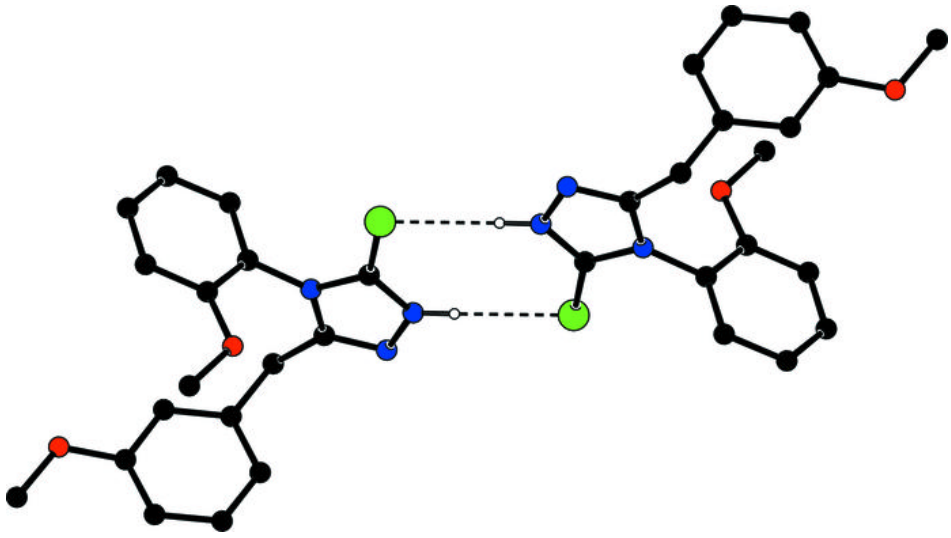
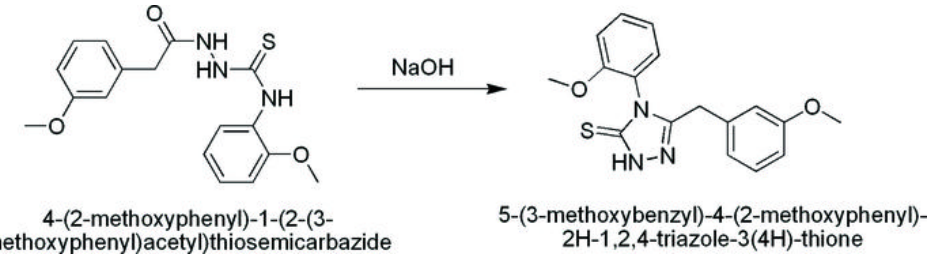


Fig. 3



4-(2-methoxyphenyl)-1-(2-(3-methoxyphenyl)acetyl)thiosemicarbazide

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