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

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.084
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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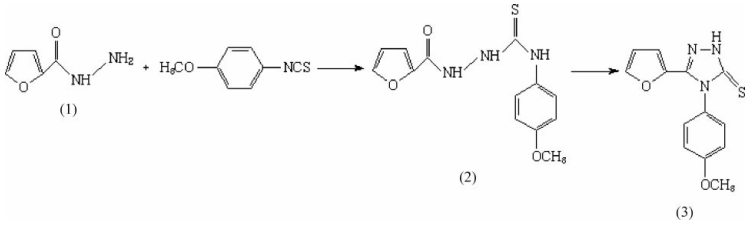
The title compound, $C_{13}H_{11}N_3O_2S$, has a non-planar conformation. The dihedral angles are 3.41 (8) and 85.48 (7)° between the triazole ring plane and the furan and benzene ring planes, respectively. The crystal packing is stabilized by several hydrogen bonds.

Comment

Derivatives of 1,2,4-triazole are known to exhibit anti-inflammatory (Unangst *et al.*, 1992; Mullican *et al.*, 1993), antiviral, analgesic (Sughen & Yoloye, 1978), antimicrobial (Shams El-Dine & Hazzaa, 1974; Misato *et al.*, 1977; Cansız *et al.*, 2001), anticonvulsant (Stillings *et al.*, 1986) and antidepressant activity (Kane *et al.*, 1988), the latter being usually explored by the forced swim test (Porsolt *et al.*, 1977; Vamvakides, 1990). Among the pharmacological profiles of 1,2,4-triazoles, their antimicrobial, anticonvulsant and antidepressant properties seem to be the best documented. The derivatives of 4,5-disubstituted 1,2,4-triazole are known to be synthesized by intramolecular cyclization of 1,4-disubstituted thiosemicarbazides (Zamani *et al.*, 2003; Cansız *et al.*, 2004; Koparır *et al.*, 2004). In addition, there are some studies on the electronic structures and thiol–thione tautomeric equilibrium of heterocyclic thione derivatives (Aydoğan *et al.*, 2002; Charistos *et al.*, 1994).



In the present study, 5-(2-furyl)-4-(4-methoxyphenyl)-2,4-dihydro-3*H*-1,2,4-triazole-3-thione, (3), was synthesized by the reaction of 1-isothiocyanato-4-methoxybenzene and 2-furohydrazide, (1), through 2-(2-furoyl)-*N*-(4-methoxyphenyl)-hydrazinecarbothioamide, (2). Base-catalysed intramolecular dehydrative cyclization of this intermediate furnished the thione in good yield (70–80%). The reaction sequences depicted in the scheme were followed to obtain the new compound. The structures of these compounds have been determined by IR and ^{1}H NMR spectra.

A perspective view of the molecule of (3) with the atomic numbering is shown in Fig. 1. In the molecular structure all the bond lengths and angles agree well with the values found by Öztürk *et al.* (2004), Akkurt *et al.* (2004) and Dege *et al.* (2004).

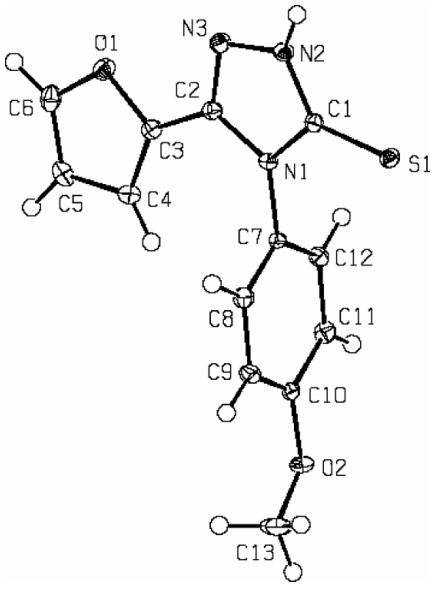


Figure 1
An *ORTEP*-3 (Farrugia, 1997) view of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

In the crystal structure, the packing of the molecules is stabilized by N—H···S and C—H···O hydrogen bonds (Table 2 and Fig. 2).

Experimental

A mixture of 2-furohydrazide, (1) (0.01 mol, 1.26 g), and 1-isothiocyanato-4-methoxybenzene (0.01 mol, 1.65 g) in absolute ethanol (100 ml) was refluxed for 8 h. The solid material obtained on cooling was filtered, washed with diethyl ether, dried and crystallized from a mixture of ethanol and acetone (75:25) (yield 93%, m.p. 435–436 K). IR (cm^{-1}): ν 3355–3320 (NH), 1678 (C═O), 1271 (C═S), 1253 (C—O—C); ^{1}H NMR: δ 3.79 (*s*, 3H, OCH_3), 5.90–6.20 (*m*, 3H, furan), 6.55–6.90 (*m*, 4H, Ar. H), 8.21–8.25 (*br*, 1H, –NH—Ar); 9.21–9.92 (*br*, 2H, 2*X*NH). Analysis calculated for $C_{13}H_{13}N_3O_3S$: C 53.60, H 4.50, N 14.42, S 11.01%; found: C 53.45, H 4.51, N 14.47, S 10.98%. A stirred mixture of compound (2) (1 mmol, 2.91 g) and sodium hydroxide (40 mg, 1 mmol, as a 2 *N* solution) was refluxed for 6 h. After cooling, the solution was acidified with hydrochloric acid and the precipitate was filtered off. The precipitate was then crystallized from a mixture of methanol and dioxane (60:40) (yield 78%, m.p. 518–519 K). IR (cm^{-1}): ν 3331–3258 (NH), 1618 (C═N), 1538, 1259, 1048, 948 (N—C═S, amide I, II, III and IV bands) (Habib *et al.*, 1997); ^{1}H NMR: δ 3.88 (*s*, 3H, OCH_3), 5.95–6.38 (*m*, 3H, furan), 7.09–7.55 (*m*, 4H, Ar. H), 14.00 (*s*, 1H, SH/NH). Analysis calculated for $C_{13}H_{11}N_3O_2S$: C 57.13, H 4.06, N 15.37, S 11.73%; found: C 57.19, H 3.99, N 15.25, S 11.79%.

Crystal data

$C_{13}H_{11}N_3O_2S$
M_r = 273.32
Monoclinic, $P2_1/c$
a = 7.0789 (6) Å
b = 8.4650 (6) Å
c = 21.4774 (13) Å
β = 96.492 (5)°
V = 1278.74 (16) Å^3
Z = 4
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 85 reflections
θ = 6–20°
μ = 0.25 mm^{-1}
T = 100 K
Prism, colorless
0.41 × 0.37 × 0.28 mm

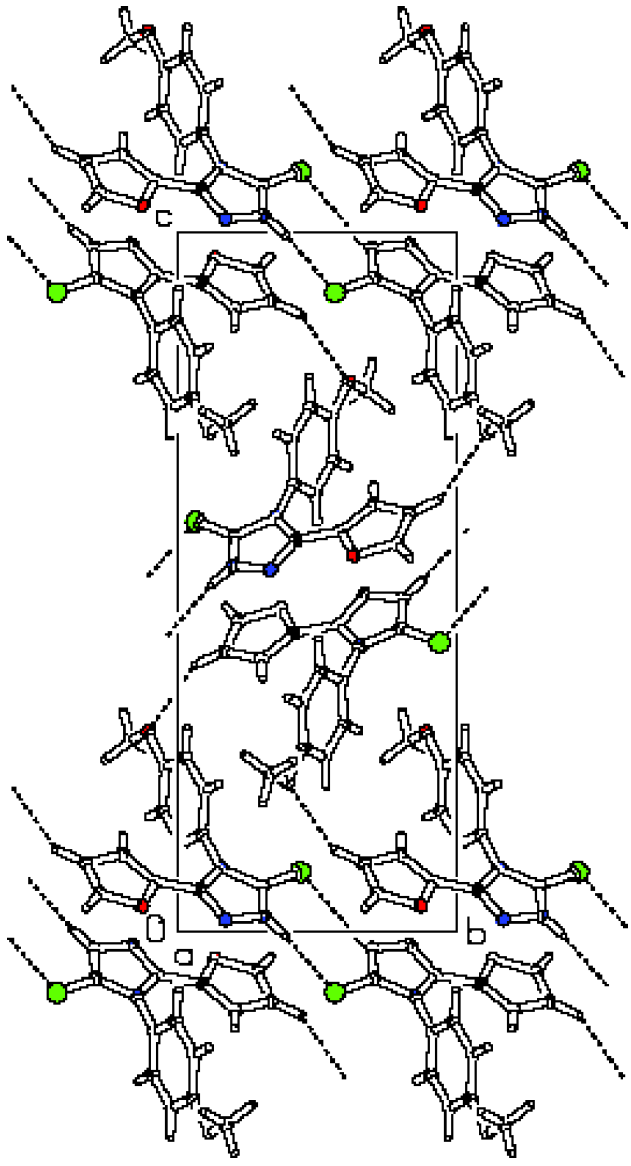


Figure 2
A view of the hydrogen bonding (dashed lines) and packing in the unit cell.

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.903, T_{max} = 0.932
18 096 measured reflections
3341 independent reflections
2719 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 29.0°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.084
S = 1.02
3341 reflections
217 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.4143P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6842 (14)	N1—C2	1.3840 (17)
O1—C3	1.3721 (16)	N1—C7	1.4377 (17)
O1—C6	1.3702 (18)	N2—N3	1.3747 (17)
O2—C10	1.3697 (16)	N2—C1	1.3394 (17)
O2—C13	1.4298 (17)	N3—C2	1.3084 (17)
N1—C1	1.3751 (17)		
C3—O1—C6	106.13 (10)	N1—C2—N3	111.08 (12)
C10—O2—C13	117.26 (11)	N1—C2—C3	124.73 (11)
C1—N1—C2	107.67 (11)	N3—C2—C3	124.18 (12)
C1—N1—C7	125.53 (11)	O1—C3—C2	114.71 (11)
C2—N1—C7	126.80 (11)	O1—C3—C4	110.34 (12)
N3—N2—C1	113.82 (11)	O1—C6—C5	110.87 (12)
N2—N3—C2	103.77 (10)	N1—C7—C8	119.35 (11)
N1—C1—N2	103.64 (11)	N1—C7—C12	119.13 (11)
S1—C1—N1	127.37 (10)	O2—C10—C11	115.57 (11)
S1—C1—N2	128.99 (11)	O2—C10—C9	123.79 (12)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.874 (17)	2.400 (17)	3.2658 (12)	170.7 (17)
C5—H5···O2^{ii}	0.993 (17)	2.510 (16)	3.4011 (17)	149.1 (12)

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

All H atoms were located in difference maps and were refined isotropically.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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