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ISSN 2414-3146

5-Methyl-1-(4-methylphenyl)-*N*′-[1-(thiophen-2-yl)ethylidene]-1*H*-1,2,3-triazole-4-carbohydrazide

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Received 26 September 2018
Accepted 28 September 2018

Edited by J. Simpson, University of Otago, New Zealand

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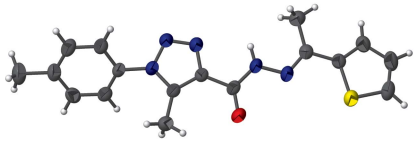
Keywords: crystal structure; thiophene; 1,2,3-triazole; π–π contacts; hydrogen bonds.

CCDC reference: 1870514

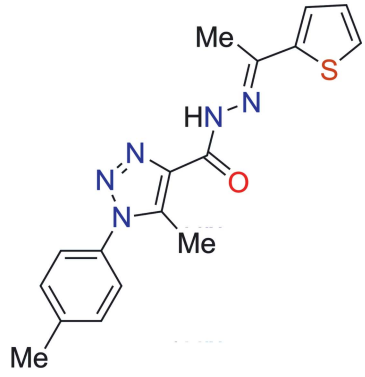
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit comprises a single molecule of $C_{17}H_{17}N_5OS$ with twist angles between the planes through the thiophenyl, methyltriazolyl and tolyl groups of 12.3 (1) and 44.9 (1)°, respectively. A possible weak intramolecular hydrogen bond forms between the methyl substituent on the triazole ring and the adjacent carbonyl O atom. In the crystal structure, π–π interactions occur between phenyl rings of pairs of molecules related by inversion symmetry with a centroid–centroid separation of 3.7647 (18) Å. The shortest intermolecular hydrogen bonding contact is a C—H···O interaction that generates inversion dimers.

3D view



Chemical scheme



Structure description

Acyl hydrazones possess diverse biological and pharmacological properties (Narang *et al.*, 2012; Popiołek 2017; Rollas & Küçükgüzel, 2007; Verma *et al.*, 2014).

The asymmetric unit comprises a molecule of $C_{17}H_{17}N_5OS$ (Fig. 1). The twist angles between the planes through the thiophenyl, methyltriazolyl and tolyl groups are 12.3 (1)° and 44.9 (1)° respectively. A weak intramolecular C10—H10*B*···O1 hydrogen bond (Table 1) imposes a degree of planarity on the section of the molecule incorporating the linked five-membered rings. In the crystal structure, inversion-related π–π interactions occur between the phenyl rings of pairs of molecules with a centroid–centroid distance $Cg3\cdots Cg3^{ii}$ = 3.7647 (18) Å, symmetry code: (ii) = 1 − *x*, 2 − *y*, −1 − *z*. Inversion-related

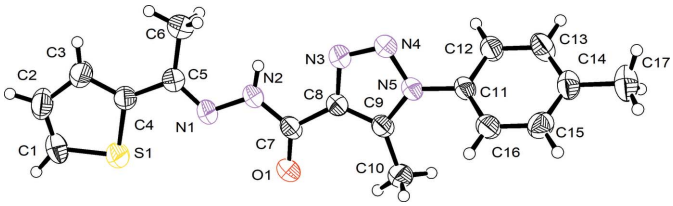


Figure 1
An *ORTEP* representation showing 50% probability ellipsoids.

C16—H16···O1 hydrogen bonds form dimers and generate $R_2^2(16)$ ring motifs (Table 1, Fig. 2).

Synthesis and crystallization

The title compound was synthesized from the reaction of a mixture of 5-methyl-1-(4-tolyl)-1*H*-1,2,3-triazole-4-carbohydrazide and 1-(thiophen-2-yl)ethanone in ethanol containing acetic acid as a catalyst under reflux conditions for 4 h. The crude product was recrystallized from dimethylformamide solution to give colourless crystals (85%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

MHA thanks King Abdulaziz City for Science and Technology (KACST), Saudi Arabia for financial support (award No. 020–0180).

References

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10*B*···O1	0.96	2.53	3.124 (4)	120
C16—H16···O1^{i}	0.93	2.55	3.437 (4)	161

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{17}N_5OS$
M_r	339.41
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	9.0292 (7), 9.864 (1), 10.8754 (9)
α, β, γ (°)	111.021 (9), 105.103 (8), 96.945 (7)
V (Å^3)	847.86 (14)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.43 × 0.18 × 0.02
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.690, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7552, 3957, 2493
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.696
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.174, 1.04
No. of reflections	3957
No. of parameters	220
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.27, −0.33

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012),*Mercury* (Macrae *et al.*, 2008) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

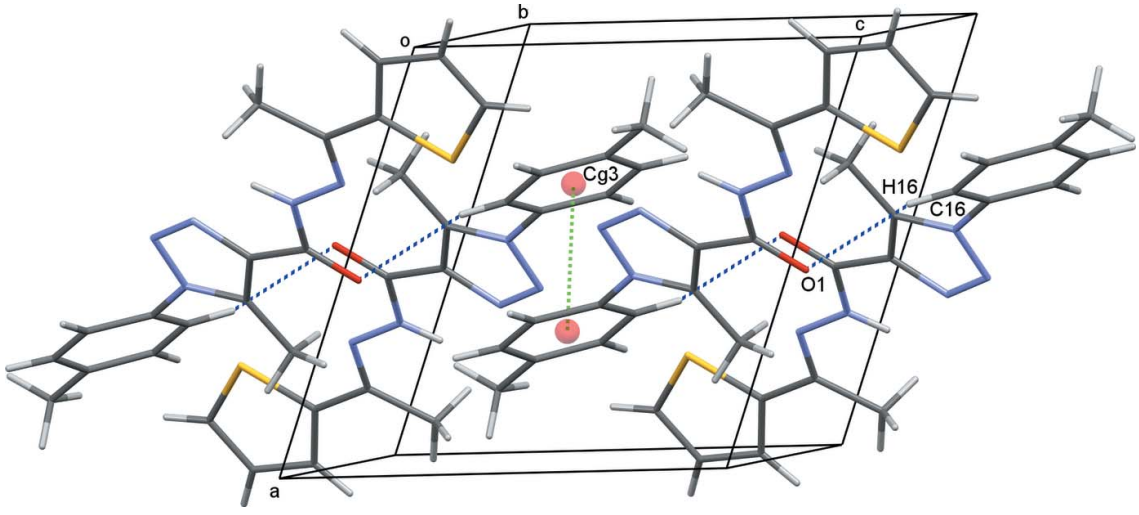

Figure 2
A view of the crystal packing approximately along the *b* axis. The π–π contact is shown as a dotted green line with the ring centroids drawn as red spheres. Hydrogen bonds are drawn as blue dotted lines.

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full crystallographic data

IUCrData (2018). **3**, x181379 [https://doi.org/10.1107/S2414314618013792]

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Crystal data

$C_{17}H_{17}N_5OS$
M_r = 339.41
Triclinic, $P\overline{1}$
a = 9.0292 (7) Å
b = 9.864 (1) Å
c = 10.8754 (9) Å
α = 111.021 (9)°
β = 105.103 (8)°
γ = 96.945 (7)°
V = 847.86 (14) Å^3
Z = 2
F(000) = 356
D_x = 1.329 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1876 reflections
θ = 4.2–26.3°
μ = 0.21 mm^{-1}
T = 296 K
Plate, colourless
0.43 × 0.18 × 0.02 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.690, T_{max} = 1.000
7552 measured reflections
3957 independent reflections
2493 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 29.6°, θ_{min} = 3.3°
h = −11→11
k = −13→11
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.174
S = 1.04
3957 reflections
220 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.2305P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1112 (3)	0.4156 (4)	0.3425 (4)	0.0601 (8)
H1	0.122378	0.438200	0.435658	0.072*
C2	−0.0111 (4)	0.3116 (4)	0.2339 (4)	0.0658 (9)
H2	−0.092623	0.254289	0.244511	0.079*
C3	−0.0012 (3)	0.2998 (3)	0.1034 (3)	0.0558 (8)
H3	−0.075280	0.233982	0.018406	0.067*
C4	0.1312 (3)	0.3974 (3)	0.1160 (3)	0.0434 (6)
C5	0.1775 (3)	0.4220 (3)	0.0050 (3)	0.0429 (6)
C6	0.0912 (3)	0.3171 (3)	−0.1446 (3)	0.0584 (8)
H6A	0.055984	0.373559	−0.197589	0.088*
H6B	0.001658	0.249516	−0.149553	0.088*
H6C	0.160803	0.261177	−0.182098	0.088*
C7	0.4505 (3)	0.6844 (3)	−0.0240 (3)	0.0421 (6)
C8	0.4679 (3)	0.7032 (3)	−0.1488 (3)	0.0404 (6)
C9	0.5722 (3)	0.8129 (3)	−0.1543 (3)	0.0399 (6)
C10	0.7022 (3)	0.9388 (3)	−0.0437 (3)	0.0539 (7)
H10A	0.791056	0.948232	−0.075463	0.081*
H10B	0.732737	0.919721	0.038836	0.081*
H10C	0.666715	1.029832	−0.022930	0.081*
C11	0.6040 (3)	0.8528 (3)	−0.3642 (3)	0.0396 (6)
C12	0.6343 (3)	0.7657 (3)	−0.4820 (3)	0.0473 (7)
H12	0.611736	0.662164	−0.513414	0.057*
C13	0.6987 (3)	0.8351 (3)	−0.5525 (3)	0.0567 (8)
H13	0.717677	0.776617	−0.632754	0.068*
C14	0.7358 (3)	0.9890 (3)	−0.5071 (3)	0.0512 (7)
C15	0.7021 (3)	1.0733 (3)	−0.3892 (3)	0.0520 (7)
H15	0.725550	1.176941	−0.357128	0.062*
C16	0.6344 (3)	1.0064 (3)	−0.3184 (3)	0.0464 (6)
H16	0.609748	1.063996	−0.241044	0.056*
C17	0.8067 (4)	1.0635 (4)	−0.5852 (4)	0.0730 (10)
H17A	0.803269	1.166931	−0.551180	0.110*
H17B	0.747381	1.014761	−0.682920	0.110*
H17C	0.914343	1.056050	−0.571143	0.110*
N1	0.2922 (2)	0.5357 (2)	0.0459 (2)	0.0451 (5)
N2	0.3362 (2)	0.5630 (2)	−0.0562 (2)	0.0473 (6)
H2A	0.290362	0.502099	−0.141972	0.057*
N3	0.3746 (2)	0.6096 (2)	−0.2804 (2)	0.0472 (6)
N4	0.4142 (3)	0.6548 (2)	−0.3689 (2)	0.0482 (6)
N5	0.5345 (2)	0.7799 (2)	−0.2928 (2)	0.0409 (5)

O1	0.5302 (2)	0.7712 (2)	0.0941 (2)	0.0595 (6)
S1	0.24161 (8)	0.49989 (8)	0.28844 (8)	0.0522 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0675 (19)	0.076 (2)	0.056 (2)	0.0198 (16)	0.0326 (16)	0.0388 (17)
C2	0.0576 (18)	0.078 (2)	0.072 (2)	0.0041 (16)	0.0295 (17)	0.0404 (19)
C3	0.0497 (15)	0.0606 (18)	0.055 (2)	0.0010 (13)	0.0169 (14)	0.0255 (16)
C4	0.0449 (14)	0.0430 (14)	0.0450 (16)	0.0106 (11)	0.0177 (12)	0.0188 (12)
C5	0.0432 (14)	0.0444 (15)	0.0435 (16)	0.0108 (11)	0.0164 (12)	0.0190 (12)
C6	0.0608 (17)	0.0615 (18)	0.0431 (18)	0.0035 (14)	0.0146 (14)	0.0157 (15)
C7	0.0494 (14)	0.0451 (15)	0.0360 (16)	0.0141 (11)	0.0199 (12)	0.0162 (12)
C8	0.0453 (13)	0.0402 (14)	0.0364 (15)	0.0092 (10)	0.0169 (11)	0.0142 (11)
C9	0.0463 (13)	0.0399 (14)	0.0350 (15)	0.0118 (11)	0.0151 (11)	0.0154 (11)
C10	0.0639 (17)	0.0500 (16)	0.0406 (17)	0.0021 (13)	0.0122 (14)	0.0175 (13)
C11	0.0453 (13)	0.0437 (14)	0.0332 (14)	0.0104 (11)	0.0143 (11)	0.0185 (12)
C12	0.0600 (16)	0.0440 (15)	0.0440 (17)	0.0136 (12)	0.0238 (13)	0.0194 (13)
C13	0.0719 (19)	0.0639 (19)	0.0479 (19)	0.0236 (15)	0.0332 (15)	0.0259 (15)
C14	0.0498 (15)	0.0613 (18)	0.0529 (19)	0.0113 (13)	0.0191 (14)	0.0337 (15)
C15	0.0564 (16)	0.0446 (16)	0.0527 (19)	0.0063 (12)	0.0109 (14)	0.0239 (14)
C16	0.0556 (15)	0.0453 (15)	0.0392 (16)	0.0126 (12)	0.0174 (13)	0.0166 (13)
C17	0.077 (2)	0.088 (2)	0.079 (3)	0.0165 (18)	0.0374 (19)	0.054 (2)
N1	0.0508 (12)	0.0516 (13)	0.0391 (13)	0.0097 (10)	0.0199 (10)	0.0225 (11)
N2	0.0565 (13)	0.0517 (13)	0.0335 (13)	0.0030 (10)	0.0177 (11)	0.0180 (11)
N3	0.0510 (13)	0.0505 (13)	0.0385 (13)	0.0037 (10)	0.0160 (11)	0.0180 (11)
N4	0.0544 (13)	0.0484 (13)	0.0357 (13)	0.0005 (10)	0.0138 (11)	0.0149 (11)
N5	0.0478 (12)	0.0430 (12)	0.0338 (12)	0.0092 (9)	0.0156 (10)	0.0169 (10)
O1	0.0732 (13)	0.0607 (13)	0.0347 (12)	−0.0031 (10)	0.0198 (10)	0.0131 (10)
S1	0.0562 (4)	0.0576 (5)	0.0451 (5)	0.0080 (3)	0.0177 (3)	0.0243 (4)

Geometric parameters (Å, º)

C1—C2	1.358 (5)	C10—H10B	0.9600
C1—S1	1.703 (3)	C10—H10C	0.9600
C1—H1	0.9300	C11—C12	1.380 (4)
C2—C3	1.410 (5)	C11—C16	1.380 (4)
C2—H2	0.9300	C11—N5	1.435 (3)
C3—C4	1.385 (4)	C12—C13	1.380 (4)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.462 (4)	C13—C14	1.384 (4)
C4—S1	1.719 (3)	C13—H13	0.9300
C5—N1	1.284 (3)	C14—C15	1.388 (4)
C5—C6	1.504 (4)	C14—C17	1.513 (4)
C6—H6A	0.9600	C15—C16	1.384 (4)
C6—H6B	0.9600	C15—H15	0.9300
C6—H6C	0.9600	C16—H16	0.9300
C7—O1	1.217 (3)	C17—H17A	0.9600

C7—N2	1.354 (3)	C17—H17B	0.9600
C7—C8	1.479 (4)	C17—H17C	0.9600
C8—N3	1.358 (3)	N1—N2	1.372 (3)
C8—C9	1.375 (3)	N2—H2A	0.8600
C9—N5	1.358 (3)	N3—N4	1.305 (3)
C9—C10	1.486 (4)	N4—N5	1.364 (3)
C10—H10A	0.9600		

C2—C1—S1	112.2 (3)	H10B—C10—H10C	109.5
C2—C1—H1	123.9	C12—C11—C16	121.0 (2)
S1—C1—H1	123.9	C12—C11—N5	118.6 (2)
C1—C2—C3	112.8 (3)	C16—C11—N5	120.4 (2)
C1—C2—H2	123.6	C13—C12—C11	118.8 (3)
C3—C2—H2	123.6	C13—C12—H12	120.6
C4—C3—C2	112.3 (3)	C11—C12—H12	120.6
C4—C3—H3	123.8	C12—C13—C14	121.9 (3)
C2—C3—H3	123.8	C12—C13—H13	119.0
C3—C4—C5	128.1 (3)	C14—C13—H13	119.0
C3—C4—S1	110.7 (2)	C13—C14—C15	117.8 (3)
C5—C4—S1	121.11 (19)	C13—C14—C17	121.3 (3)
N1—C5—C4	115.4 (2)	C15—C14—C17	120.8 (3)
N1—C5—C6	125.0 (3)	C16—C15—C14	121.4 (3)
C4—C5—C6	119.6 (2)	C16—C15—H15	119.3
C5—C6—H6A	109.5	C14—C15—H15	119.3
C5—C6—H6B	109.5	C11—C16—C15	119.0 (3)
H6A—C6—H6B	109.5	C11—C16—H16	120.5
C5—C6—H6C	109.5	C15—C16—H16	120.5
H6A—C6—H6C	109.5	C14—C17—H17A	109.5
H6B—C6—H6C	109.5	C14—C17—H17B	109.5
O1—C7—N2	124.6 (3)	H17A—C17—H17B	109.5
O1—C7—C8	122.6 (2)	C14—C17—H17C	109.5
N2—C7—C8	112.8 (2)	H17A—C17—H17C	109.5
N3—C8—C9	109.3 (2)	H17B—C17—H17C	109.5
N3—C8—C7	122.5 (2)	C5—N1—N2	116.6 (2)
C9—C8—C7	128.1 (2)	C7—N2—N1	121.1 (2)
N5—C9—C8	103.6 (2)	C7—N2—H2A	119.5
N5—C9—C10	124.4 (2)	N1—N2—H2A	119.5
C8—C9—C10	131.9 (3)	N4—N3—C8	109.2 (2)
C9—C10—H10A	109.5	N3—N4—N5	107.0 (2)
C9—C10—H10B	109.5	C9—N5—N4	110.9 (2)
H10A—C10—H10B	109.5	C9—N5—C11	129.9 (2)
C9—C10—H10C	109.5	N4—N5—C11	119.1 (2)
H10A—C10—H10C	109.5	C1—S1—C4	92.00 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O1	0.96	2.53	3.124 (4)	120

C16—H16···O1^{i}	0.93	2.55	3.437 (4)	161

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