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3-(4-Amino-3-methyl-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-yl)-3-(2-furyl)-1-phenylpropan-1-one

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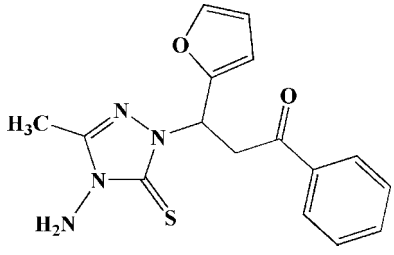
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.049; wR factor = 0.119; data-to-parameter ratio = 17.3.

In the title molecule, $C_{16}H_{16}N_4O_2S$, the plane of the 1,2,4-triazole ring forms dihedral angles of 77.9 (2) and 30.0 (2)° with the planes of the furyl and phenyl rings, respectively. Weak intermolecular N—H···S and C—H···O hydrogen bonds consolidate the crystal packing.

Related literature

For the crystal structures of related 1,2,4-triazole-5(4*H*)-thione derivates, see: Al-Tamimi *et al.* (2010); Fun *et al.* (2009); Tan *et al.* (2010); Wang *et al.* (2011).



Experimental

Crystal data

$C_{16}H_{16}N_4O_2S$
M_r = 328.39
Monoclinic, $P2_1/n$
a = 7.3702 (8) Å
b = 24.131 (2) Å
c = 9.240 (1) Å
β = 106.745 (5)°
V = 1573.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.12 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.957, T_{max} = 0.974
15993 measured reflections
3746 independent reflections
2835 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
S = 1.06
3746 reflections
217 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···S1^{i}	0.95 (3)	2.64 (3)	3.475 (2)	148 (2)
C4—H4D···O1^{ii}	0.99	2.45	3.346 (2)	151
C14—H14···O1^{iii}	0.95	2.55	3.490 (2)	169

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5087).

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supporting information

Acta Cryst. (2011). E**67**, o1609 [doi:10.1107/S1600536811020988]

3-(4-Amino-3-methyl-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-yl)-3-(2-furyl)-1-phenylpropan-1-one

Yan Gao, Lu Wang and He-wen Wang

S1. Comment

In continuation of structural study of 1,2,4-triazole-5(4*H*)-thione derivatives in our group (Wang *et al.*, 2011), we present here the crystal structure of the title compound, (I).

In (I) (Fig. 1), the bond lengths and angles are normal and comparable with those reported for the related 1,2,4-triazole-5(4*H*)- thione derivatives (Al-Tamimi *et al.*, 2010; Fun *et al.*, 2009; Tan *et al.*, 2010; Wang *et al.*, 2011). The 1,2,4-triazole ring makes the dihedral angles of 30.2 (2) and 77.9 (2)° with the phenyl ring and the furanyl ring, respectively. The phenyl and furanyl rings form a dihedral angle of 71.1 (2) °.

Intermolecular N—H···S hydrogen bond (Table 1) links the adjacent molecule into centrosymmetric dimers, which are further linked by the weak C—H···O interactions (Table 1) into three-dimensional structure.

S2. Experimental

The title compound was synthesized by the reaction of the 3-(furan-2-yl)-1- phenyl-2-propen-1-one (2.0 mmol) with 4-amino-3-methyl-4*H*-1,2,4-triazole-5- thiol (2.0 mmol) in ethanol. The reaction progress was monitored *via* TLC. The resulting precipitate was filtered off, washed with cold ethanol, dried and purified to give the target product as colorless solid in 84% yield. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform-ethanol (1:1).

S3. Refinement

N-bound H atoms were located in a difference map and isotropically refined. C-bound H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding, with U_{iso}(H) = 1.2-1.5 U_{eq}(C).

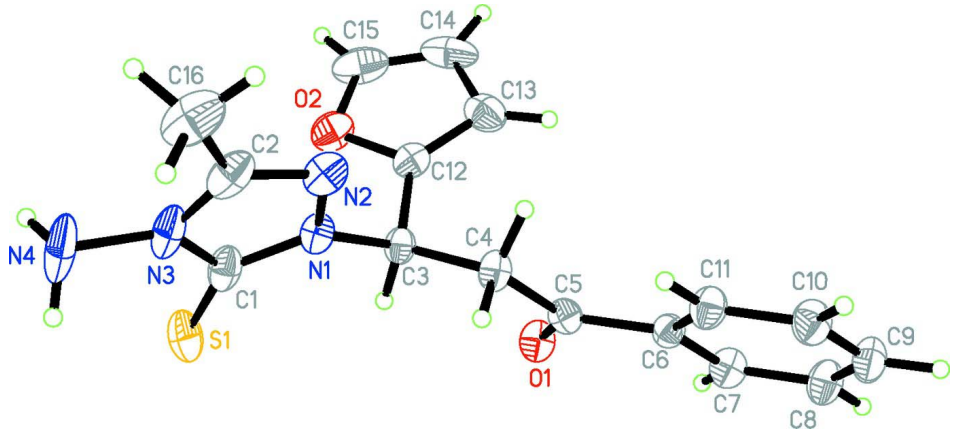

Figure 1

View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 55% probability level.

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$c = 9.240\ (1)$ Å
$\beta = 106.745\ (5)°$
$V = 1573.7\ (3)$ Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4962 reflections
$\theta = 1.7–27.9°$
$\mu = 0.22$ mm^{-1}
$T = 113$ K
Prism, colorless
0.20 × 0.18 × 0.12 mm

Data collection

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Radiation source: rotating anode
Multilayer monochromator
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φ and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.957$, $T_{max} = 0.974$
15993 measured reflections
3746 independent reflections
2835 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 27.9°$, $\theta_{min} = 1.7°$
$h = -9\rightarrow9$
$k = -31\rightarrow31$
$l = -12\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.119$
$S = 1.06$
3746 reflections
217 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.32564 (7)	0.044074 (19)	0.12279 (6)	0.03395 (17)
O1	0.44017 (17)	0.24184 (5)	0.30582 (14)	0.0252 (3)
O2	0.76075 (17)	0.12393 (5)	0.14479 (15)	0.0302 (3)
N1	0.3805 (2)	0.13261 (6)	−0.04619 (17)	0.0216 (3)
N2	0.3656 (2)	0.14722 (6)	−0.19396 (18)	0.0266 (4)
N3	0.2807 (2)	0.06078 (6)	−0.17722 (18)	0.0298 (4)
N4	0.2143 (3)	0.00728 (7)	−0.2257 (2)	0.0447 (6)
H4A	0.312 (4)	−0.0188 (10)	−0.184 (3)	0.065 (8)*
H4B	0.120 (4)	0.0015 (10)	−0.184 (3)	0.054 (8)*
C1	0.3301 (2)	0.07965 (7)	−0.0317 (2)	0.0256 (4)
C2	0.3022 (3)	0.10221 (8)	−0.2710 (2)	0.0304 (5)
C3	0.4564 (2)	0.17151 (6)	0.0778 (2)	0.0196 (4)
H3	0.4213	0.1574	0.1679	0.023*
C4	0.3677 (2)	0.22862 (6)	0.0401 (2)	0.0219 (4)
H4C	0.4291	0.2481	−0.0277	0.026*
H4D	0.2315	0.2244	−0.0144	0.026*
C5	0.3886 (2)	0.26319 (7)	0.1805 (2)	0.0204 (4)
C6	0.3390 (2)	0.32323 (7)	0.1632 (2)	0.0199 (4)
C7	0.3636 (3)	0.35495 (7)	0.2932 (2)	0.0255 (4)
H7	0.4096	0.3380	0.3897	0.031*
C8	0.3215 (3)	0.41086 (7)	0.2828 (2)	0.0298 (5)
H8	0.3391	0.4324	0.3718	0.036*
C9	0.2533 (3)	0.43531 (7)	0.1412 (2)	0.0297 (5)
H9	0.2260	0.4738	0.1338	0.036*
C10	0.2250 (3)	0.40402 (7)	0.0111 (2)	0.0289 (4)
H10	0.1765	0.4209	−0.0852	0.035*
C11	0.2673 (3)	0.34799 (7)	0.0217 (2)	0.0251 (4)
H11	0.2475	0.3264	−0.0675	0.030*
C12	0.6687 (2)	0.17414 (7)	0.1194 (2)	0.0222 (4)
C13	0.7944 (3)	0.21587 (8)	0.1527 (2)	0.0310 (5)
H13	0.7673	0.2544	0.1447	0.037*
C14	0.9781 (3)	0.19038 (10)	0.2031 (2)	0.0388 (5)
H14	1.0966	0.2089	0.2356	0.047*
C15	0.9504 (3)	0.13569 (10)	0.1956 (2)	0.0384 (5)
H15	1.0483	0.1087	0.2219	0.046*

C16	0.2566 (3)	0.09657 (10)	−0.4376 (2)	0.0435 (6)
H16A	0.2883	0.1311	−0.4806	0.065*
H16B	0.1211	0.0889	−0.4801	0.065*
H16C	0.3303	0.0660	−0.4620	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0401 (3)	0.0202 (2)	0.0366 (3)	−0.00212 (19)	0.0030 (2)	0.0041 (2)
O1	0.0304 (7)	0.0238 (6)	0.0186 (7)	0.0022 (5)	0.0025 (5)	0.0014 (5)
O2	0.0255 (7)	0.0342 (7)	0.0290 (8)	0.0083 (5)	0.0051 (6)	0.0028 (6)
N1	0.0240 (8)	0.0204 (7)	0.0175 (9)	0.0025 (6)	0.0014 (6)	−0.0010 (6)
N2	0.0261 (8)	0.0324 (8)	0.0186 (9)	0.0048 (6)	0.0019 (7)	−0.0012 (7)
N3	0.0307 (9)	0.0222 (7)	0.0295 (10)	0.0064 (6)	−0.0028 (7)	−0.0095 (7)
N4	0.0461 (12)	0.0232 (8)	0.0509 (14)	0.0068 (8)	−0.0081 (11)	−0.0183 (9)
C1	0.0226 (9)	0.0205 (8)	0.0280 (11)	0.0051 (7)	−0.0017 (8)	−0.0039 (8)
C2	0.0243 (10)	0.0376 (10)	0.0247 (11)	0.0106 (8)	−0.0003 (8)	−0.0070 (9)
C3	0.0227 (9)	0.0179 (8)	0.0162 (10)	−0.0002 (7)	0.0027 (7)	−0.0023 (7)
C4	0.0260 (9)	0.0190 (8)	0.0180 (10)	0.0021 (7)	0.0021 (7)	0.0004 (7)
C5	0.0182 (8)	0.0217 (8)	0.0191 (10)	−0.0022 (7)	0.0015 (7)	−0.0008 (7)
C6	0.0184 (8)	0.0192 (8)	0.0212 (10)	−0.0006 (6)	0.0043 (7)	−0.0018 (7)
C7	0.0279 (10)	0.0268 (9)	0.0199 (10)	0.0026 (7)	0.0036 (8)	0.0013 (8)
C8	0.0361 (11)	0.0248 (9)	0.0272 (11)	0.0018 (8)	0.0070 (9)	−0.0055 (8)
C9	0.0374 (11)	0.0202 (9)	0.0320 (12)	0.0033 (8)	0.0106 (9)	−0.0013 (8)
C10	0.0366 (11)	0.0262 (9)	0.0238 (11)	0.0046 (8)	0.0087 (8)	0.0060 (8)
C11	0.0302 (10)	0.0235 (8)	0.0205 (11)	0.0014 (7)	0.0056 (8)	−0.0026 (7)
C12	0.0232 (9)	0.0259 (9)	0.0168 (10)	0.0034 (7)	0.0045 (7)	0.0011 (7)
C13	0.0342 (11)	0.0360 (10)	0.0243 (11)	−0.0091 (8)	0.0105 (9)	−0.0037 (8)
C14	0.0229 (10)	0.0715 (15)	0.0229 (11)	−0.0114 (10)	0.0079 (8)	−0.0037 (11)
C15	0.0207 (10)	0.0649 (15)	0.0277 (12)	0.0103 (10)	0.0038 (8)	0.0039 (11)
C16	0.0357 (12)	0.0648 (14)	0.0250 (13)	0.0099 (10)	0.0008 (9)	−0.0121 (11)

Geometric parameters (Å, º)

S1—C1	1.674 (2)	C6—C7	1.391 (2)
O1—C5	1.223 (2)	C6—C11	1.396 (2)
O2—C15	1.370 (2)	C7—C8	1.381 (2)
O2—C12	1.3755 (19)	C7—H7	0.9500
N1—C1	1.348 (2)	C8—C9	1.390 (3)
N1—N2	1.383 (2)	C8—H8	0.9500
N1—C3	1.462 (2)	C9—C10	1.383 (3)
N2—C2	1.309 (2)	C9—H9	0.9500
N3—C2	1.362 (3)	C10—C11	1.385 (2)
N3—C1	1.366 (2)	C10—H10	0.9500
N3—N4	1.407 (2)	C11—H11	0.9500
N4—H4A	0.95 (3)	C12—C13	1.343 (2)
N4—H4B	0.89 (2)	C13—C14	1.437 (3)
C2—C16	1.484 (3)	C13—H13	0.9500

C3—C12	1.501 (2)	C14—C15	1.334 (3)
C3—C4	1.522 (2)	C14—H14	0.9500
C3—H3	1.0000	C15—H15	0.9500
C4—C5	1.513 (2)	C16—H16A	0.9800
C4—H4C	0.9900	C16—H16B	0.9800
C4—H4D	0.9900	C16—H16C	0.9800
C5—C6	1.491 (2)		

C15—O2—C12	106.30 (14)	C11—C6—C5	122.09 (16)
C1—N1—N2	113.18 (14)	C8—C7—C6	120.44 (17)
C1—N1—C3	125.81 (15)	C8—C7—H7	119.8
N2—N1—C3	120.89 (14)	C6—C7—H7	119.8
C2—N2—N1	103.86 (15)	C7—C8—C9	119.52 (18)
C2—N3—C1	109.54 (15)	C7—C8—H8	120.2
C2—N3—N4	124.36 (17)	C9—C8—H8	120.2
C1—N3—N4	126.09 (18)	C10—C9—C8	120.61 (16)
N3—N4—H4A	109.0 (14)	C10—C9—H9	119.7
N3—N4—H4B	104.4 (16)	C8—C9—H9	119.7
H4A—N4—H4B	108 (2)	C9—C10—C11	119.81 (17)
N1—C1—N3	102.79 (16)	C9—C10—H10	120.1
N1—C1—S1	130.11 (14)	C11—C10—H10	120.1
N3—C1—S1	127.09 (14)	C10—C11—C6	120.04 (17)
N2—C2—N3	110.62 (17)	C10—C11—H11	120.0
N2—C2—C16	125.36 (19)	C6—C11—H11	120.0
N3—C2—C16	124.02 (17)	C13—C12—O2	110.33 (16)
N1—C3—C12	111.29 (14)	C13—C12—C3	133.62 (16)
N1—C3—C4	111.10 (13)	O2—C12—C3	115.67 (14)
C12—C3—C4	111.56 (13)	C12—C13—C14	106.08 (18)
N1—C3—H3	107.6	C12—C13—H13	127.0
C12—C3—H3	107.6	C14—C13—H13	127.0
C4—C3—H3	107.6	C15—C14—C13	106.89 (18)
C5—C4—C3	111.85 (14)	C15—C14—H14	126.6
C5—C4—H4C	109.2	C13—C14—H14	126.6
C3—C4—H4C	109.2	C14—C15—O2	110.40 (17)
C5—C4—H4D	109.2	C14—C15—H15	124.8
C3—C4—H4D	109.2	O2—C15—H15	124.8
H4C—C4—H4D	107.9	C2—C16—H16A	109.5
O1—C5—C6	120.69 (16)	C2—C16—H16B	109.5
O1—C5—C4	120.43 (15)	H16A—C16—H16B	109.5
C6—C5—C4	118.85 (15)	C2—C16—H16C	109.5
C7—C6—C11	119.55 (15)	H16A—C16—H16C	109.5
C7—C6—C5	118.35 (16)	H16B—C16—H16C	109.5

C1—N1—N2—C2	0.83 (19)	O1—C5—C6—C7	3.7 (2)
C3—N1—N2—C2	177.05 (14)	C4—C5—C6—C7	−178.26 (15)
N2—N1—C1—N3	−0.38 (19)	O1—C5—C6—C11	−175.51 (16)
C3—N1—C1—N3	−176.37 (15)	C4—C5—C6—C11	2.5 (2)
N2—N1—C1—S1	179.91 (13)	C11—C6—C7—C8	−1.4 (3)

C3—N1—C1—S1	3.9 (3)	C5—C6—C7—C8	179.31 (16)
C2—N3—C1—N1	−0.21 (19)	C6—C7—C8—C9	0.3 (3)
N4—N3—C1—N1	−179.21 (17)	C7—C8—C9—C10	0.9 (3)
C2—N3—C1—S1	179.51 (14)	C8—C9—C10—C11	−0.9 (3)
N4—N3—C1—S1	0.5 (3)	C9—C10—C11—C6	−0.2 (3)
N1—N2—C2—N3	−0.94 (19)	C7—C6—C11—C10	1.4 (3)
N1—N2—C2—C16	178.24 (17)	C5—C6—C11—C10	−179.39 (16)
C1—N3—C2—N2	0.8 (2)	C15—O2—C12—C13	0.0 (2)
N4—N3—C2—N2	179.78 (17)	C15—O2—C12—C3	−173.96 (16)
C1—N3—C2—C16	−178.42 (17)	N1—C3—C12—C13	137.1 (2)
N4—N3—C2—C16	0.6 (3)	C4—C3—C12—C13	12.4 (3)
C1—N1—C3—C12	97.51 (19)	N1—C3—C12—O2	−50.8 (2)
N2—N1—C3—C12	−78.20 (18)	C4—C3—C12—O2	−175.52 (15)
C1—N1—C3—C4	−137.52 (17)	O2—C12—C13—C14	−0.2 (2)
N2—N1—C3—C4	46.8 (2)	C3—C12—C13—C14	172.20 (19)
N1—C3—C4—C5	159.98 (14)	C12—C13—C14—C15	0.4 (2)
C12—C3—C4—C5	−75.20 (19)	C13—C14—C15—O2	−0.4 (2)
C3—C4—C5—O1	−12.1 (2)	C12—O2—C15—C14	0.3 (2)
C3—C4—C5—C6	169.87 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···S1^{i}	0.95 (3)	2.64 (3)	3.475 (2)	148 (2)
C4—H4*D*···O1^{ii}	0.99	2.45	3.346 (2)	151
C14—H14···O1^{iii}	0.95	2.55	3.490 (2)	169

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