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1-[2-Imino-4-methyl-3-(4-nitrophenyl)-2,3-dihydrothiazol-5-yl]ethanone

Yan-Fang Lin, Guang-Xiang Zhong, Feng Xu and Wei-Xiao Hu*

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310032, People's Republic of China
Correspondence e-mail: huyang@mail.hz.zj.cn

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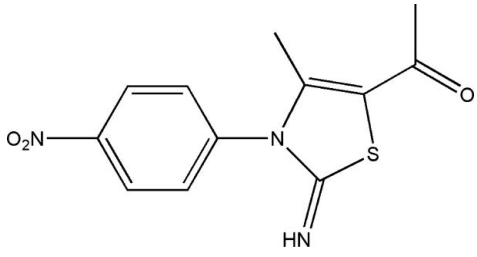
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.110; data-to-parameter ratio = 16.6.

The title compound, $C_{12}H_{11}N_3O_3S$, was obtained as a by-product during the attempted preparation of a thiazolone. The dihedral angle between the benzene and the thiazole ring is 85.36 (5)°. The ethanone group is coplanar with the thiazole ring, whereas the nitro group is twisted away from the attached benzene ring by 10.7 (2)°. Intermolecular C—H···O and N—H···O hydrogen bonds, involving the ethanone O atom, link the molecules into a two-dimensional network parallel to the *bc* plane.

Related literature

For synthesis of thiazolone, see: Patil *et al.* (1978).



Experimental

Crystal data

$C_{12}H_{11}N_3O_3S$
M_r = 277.30
Monoclinic, $P2_1/c$
a = 11.446 (2) Å
b = 11.1475 (19) Å
c = 10.5155 (18) Å
β = 103.108 (2)°
V = 1306.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 296 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.939, T_{max} = 0.941
8104 measured reflections
2957 independent reflections
2218 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.110
S = 1.03
2957 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O1^{i}	0.85 (2)	2.39 (2)	3.174 (2)	153 (2)
C8—H8···O1^{ii}	0.93	2.29	3.209 (2)	168
C15—H15B···O1^{iii}	0.96	2.49	3.434 (2)	166

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2428).

References

Bruker (2005). *APEX2*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Patil, V. H., Mane, R. A. & Ingle, D. B. (1978). *Indian J. Chem.* Sect. B, **16**, 1114–1116.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o3699 [**doi:10.1107/S1600536807037543**]

1-[2-Imino-4-methyl-3-(4-nitrophenyl)-2,3-dihydrothiazol-5-yl]ethanone

Y.-F. Lin, G.-X. Zhong, F. Xu and W.-X. Hu

Comment

The title compound, (I), was obtained as a by-product during the attempted preparation of a thiazolone (Patil *et al.*, 1978). We report here the crystal structure of (I).

The molecule structure of (I) is illustrated in Fig. 1. The benzene ring is twisted out of the central thiazole ring by 85.36 (5)°. The ethanone group is coplanar with the thiazole ring [dihedral angle = 2.6 (1)°] whereas the nitro group forms a dihedral angle of 10.7 (2)° with the attached benzene ring.

In the crystal structure, weak intermolecular C—H···O and N—H···O hydrogen bonds (Table 1), involving the ethanone oxygen atom, link the molecules into a two-dimensional network parallel to the *bc* plane (Fig. 2).

Experimental

A mixture of 3-bromopentane-2,4-dione (1.79 g, 0.01 mol) and 1-(4-nitrophenyl)thiourea (1.97 g, 0.01 mol) in acetone (20 ml) was refluxed for 1 h. The mixture was cooled to room temperature, then it was neutralized with 15% sodium carbonate aqueous solution to a pH of 8, to obtain a yellow precipitate. The precipitate was filtered off and recrystallized from acetone to give the crude product. The solid product was dissolved in ethanol evaporated gradually at room temperature to afford single crystals of the title compound (m.p. 441–441.5 K). ^{1}HNMR($CDCl_3$)σp.p.m.: 8.42(d,2*H*,Ar), 7.50(d,2*H*,Ar), 2.36(s,1*H*,CH_3), 2.25(s,3*H*,CH_3). MS.(m/*z*,%): 276(M^+,100), 262 (15), 230 (20), 163 (80), 117 (55), 76 (30).

Refinement

Atom H6 was located in a difference Fourier map and refined isotropically with the N—H bond restraint of 0.86 (2) Å. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined to fit the electron density [U_{iso}(H) = 1.5U_{eq}(C)]. Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

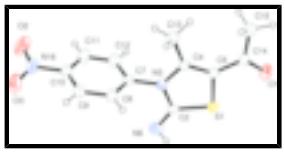


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids.

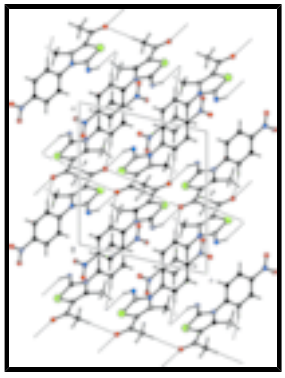


Fig. 2. The crystal structure of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

1-[2-Imino-4-methyl-3-(4-nitrophenyl)-2,3-dihydrothiazol-5-yl]ethanone

Crystal data

$C_{12}H_{11}N_3O_3S$	F_{000} = 576
M_r = 277.30	D_x = 1.409 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2619 reflections
a = 11.446 (2) Å	θ = 2.6–26.9º
b = 11.1475 (19) Å	μ = 0.26 mm^{-1}
c = 10.5155 (18) Å	T = 296 (2) K
β = 103.108 (2)º	Prism, colourless
V = 1306.8 (4) Å^3	0.25 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX II CCD area-detector diffractometer	2957 independent reflections
Radiation source: fine-focus sealed tube	2218 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −14→12
T_{min} = 0.939, T_{max} = 0.941	k = −14→14
8104 measured reflections	l = −13→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.038	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.1727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2957 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
178 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.41478 (4)	0.47752 (4)	0.66509 (4)	0.05007 (16)
C2	0.29327 (16)	0.54805 (15)	0.55964 (15)	0.0474 (4)
N3	0.25911 (13)	0.47702 (12)	0.44709 (13)	0.0454 (3)
C4	0.32876 (14)	0.37655 (14)	0.44183 (14)	0.0419 (4)
C5	0.41624 (14)	0.36138 (14)	0.55179 (14)	0.0423 (4)
N6	0.24008 (16)	0.64488 (15)	0.57377 (17)	0.0654 (4)
H6	0.271 (2)	0.677 (2)	0.6472 (18)	0.089 (8)*
C7	0.15721 (15)	0.51090 (14)	0.34560 (16)	0.0452 (4)
C8	0.17215 (16)	0.59002 (17)	0.25130 (18)	0.0588 (5)
H8	0.2475	0.6223	0.2534	0.071*
C9	0.07504 (17)	0.62214 (17)	0.15259 (19)	0.0606 (5)
H9	0.0837	0.6769	0.0886	0.073*
C10	−0.03356 (15)	0.57147 (17)	0.15162 (18)	0.0548 (4)
C11	−0.05053 (18)	0.4921 (2)	0.2445 (2)	0.0701 (6)
H11	−0.1257	0.4589	0.2410	0.084*
C12	0.04649 (18)	0.46203 (19)	0.3440 (2)	0.0646 (5)
H12	0.0370	0.4092	0.4093	0.078*
C13	0.30315 (17)	0.30545 (17)	0.31897 (16)	0.0571 (5)
H13A	0.3336	0.3471	0.2535	0.086*
H13B	0.2181	0.2949	0.2893	0.086*
H13C	0.3412	0.2284	0.3348	0.086*
C14	0.50708 (14)	0.27027 (15)	0.59324 (15)	0.0450 (4)
C15	0.52307 (18)	0.16729 (18)	0.50871 (18)	0.0623 (5)
H15A	0.5914	0.1210	0.5514	0.093*

H15B	0.5354	0.1968	0.4270	0.093*
H15C	0.4527	0.1177	0.4932	0.093*
N16	−0.13660 (16)	0.60437 (19)	0.04487 (18)	0.0728 (5)
O1	0.57196 (11)	0.27919 (12)	0.70342 (11)	0.0597 (3)
O2	−0.22880 (14)	0.5470 (2)	0.0333 (2)	0.1050 (6)
O3	−0.12451 (16)	0.6876 (2)	−0.02542 (17)	0.1007 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0592 (3)	0.0519 (3)	0.0348 (2)	−0.00628 (19)	0.00156 (18)	−0.00343 (16)
C2	0.0545 (10)	0.0450 (9)	0.0408 (8)	−0.0065 (7)	0.0066 (7)	−0.0018 (7)
N3	0.0502 (8)	0.0432 (7)	0.0383 (7)	0.0004 (6)	0.0004 (6)	−0.0012 (5)
C4	0.0466 (9)	0.0424 (8)	0.0352 (8)	−0.0016 (7)	0.0060 (6)	0.0009 (6)
C5	0.0474 (9)	0.0453 (8)	0.0328 (7)	−0.0059 (7)	0.0062 (6)	0.0006 (6)
N6	0.0786 (12)	0.0523 (10)	0.0596 (10)	0.0051 (8)	0.0038 (9)	−0.0137 (8)
C7	0.0458 (9)	0.0431 (8)	0.0427 (8)	0.0009 (7)	0.0017 (7)	−0.0014 (6)
C8	0.0470 (10)	0.0600 (11)	0.0622 (11)	−0.0121 (8)	−0.0030 (8)	0.0143 (9)
C9	0.0577 (11)	0.0585 (11)	0.0584 (11)	−0.0020 (9)	−0.0016 (9)	0.0127 (9)
C10	0.0447 (9)	0.0580 (10)	0.0548 (10)	0.0081 (8)	−0.0029 (8)	−0.0104 (8)
C11	0.0416 (10)	0.0887 (15)	0.0775 (14)	−0.0101 (10)	0.0082 (10)	0.0033 (11)
C12	0.0563 (11)	0.0737 (13)	0.0631 (12)	−0.0104 (10)	0.0117 (9)	0.0119 (10)
C13	0.0647 (11)	0.0601 (11)	0.0396 (9)	0.0086 (9)	−0.0027 (8)	−0.0083 (8)
C14	0.0407 (8)	0.0554 (10)	0.0374 (8)	−0.0043 (7)	0.0060 (7)	0.0063 (7)
C15	0.0595 (11)	0.0651 (11)	0.0567 (11)	0.0144 (9)	0.0012 (9)	−0.0005 (9)
N16	0.0548 (11)	0.0859 (13)	0.0671 (11)	0.0176 (9)	−0.0084 (8)	−0.0193 (10)
O1	0.0527 (7)	0.0779 (9)	0.0418 (6)	0.0014 (6)	−0.0036 (5)	0.0048 (6)
O2	0.0463 (9)	0.1384 (17)	0.1146 (14)	0.0024 (10)	−0.0146 (9)	−0.0233 (12)
O3	0.0961 (13)	0.1092 (14)	0.0755 (11)	0.0222 (11)	−0.0251 (9)	0.0102 (10)

Geometric parameters (Å, °)

S1—C2	1.7558 (18)	C10—C11	1.364 (3)
S1—C5	1.7620 (16)	C10—N16	1.478 (2)
C2—N6	1.264 (2)	C11—C12	1.383 (3)
C2—N3	1.404 (2)	C11—H11	0.93
N3—C4	1.383 (2)	C12—H12	0.93
N3—C7	1.441 (2)	C13—H13A	0.96
C4—C5	1.358 (2)	C13—H13B	0.96
C4—C13	1.487 (2)	C13—H13C	0.96
C5—C14	1.448 (2)	C14—O1	1.2303 (19)
N6—H6	0.85 (2)	C14—C15	1.488 (2)
C7—C8	1.367 (2)	C15—H15A	0.96
C7—C12	1.376 (3)	C15—H15B	0.96
C8—C9	1.385 (2)	C15—H15C	0.96
C8—H8	0.93	N16—O3	1.214 (3)
C9—C10	1.363 (3)	N16—O2	1.216 (3)
C9—H9	0.93		

C2—S1—C5	91.70 (8)	C11—C10—N16	119.07 (18)
N6—C2—N3	122.08 (16)	C10—C11—C12	118.58 (18)
N6—C2—S1	129.91 (14)	C10—C11—H11	120.7
N3—C2—S1	108.00 (12)	C12—C11—H11	120.7
C4—N3—C2	115.95 (13)	C7—C12—C11	119.52 (18)
C4—N3—C7	124.02 (13)	C7—C12—H12	120.2
C2—N3—C7	120.02 (13)	C11—C12—H12	120.2
C5—C4—N3	112.78 (13)	C4—C13—H13A	109.5
C5—C4—C13	129.46 (15)	C4—C13—H13B	109.5
N3—C4—C13	117.68 (14)	H13A—C13—H13B	109.5
C4—C5—C14	133.17 (15)	C4—C13—H13C	109.5
C4—C5—S1	111.48 (12)	H13A—C13—H13C	109.5
C14—C5—S1	115.34 (11)	H13B—C13—H13C	109.5
C2—N6—H6	110.6 (17)	O1—C14—C5	117.75 (15)
C8—C7—C12	120.87 (16)	O1—C14—C15	119.67 (16)
C8—C7—N3	119.53 (15)	C5—C14—C15	122.57 (14)
C12—C7—N3	119.59 (16)	C14—C15—H15A	109.5
C7—C8—C9	119.96 (17)	C14—C15—H15B	109.5
C7—C8—H8	120.0	H15A—C15—H15B	109.5
C9—C8—H8	120.0	C14—C15—H15C	109.5
C10—C9—C8	118.30 (17)	H15A—C15—H15C	109.5
C10—C9—H9	120.9	H15B—C15—H15C	109.5
C8—C9—H9	120.9	O3—N16—O2	123.9 (2)
C9—C10—C11	122.76 (17)	O3—N16—C10	118.08 (19)
C9—C10—N16	118.17 (19)	O2—N16—C10	118.0 (2)
C5—S1—C2—N6	−178.16 (19)	C2—N3—C7—C12	−95.0 (2)
C5—S1—C2—N3	1.87 (12)	C12—C7—C8—C9	0.1 (3)
N6—C2—N3—C4	176.94 (16)	N3—C7—C8—C9	179.31 (16)
S1—C2—N3—C4	−3.09 (17)	C7—C8—C9—C10	−1.0 (3)
N6—C2—N3—C7	−2.8 (3)	C8—C9—C10—C11	0.9 (3)
S1—C2—N3—C7	177.17 (12)	C8—C9—C10—N16	−178.98 (17)
C2—N3—C4—C5	3.0 (2)	C9—C10—C11—C12	0.2 (3)
C7—N3—C4—C5	−177.31 (14)	N16—C10—C11—C12	−179.94 (19)
C2—N3—C4—C13	−174.16 (15)	C8—C7—C12—C11	1.0 (3)
C7—N3—C4—C13	5.6 (2)	N3—C7—C12—C11	−178.22 (17)
N3—C4—C5—C14	177.98 (16)	C10—C11—C12—C7	−1.1 (3)
C13—C4—C5—C14	−5.3 (3)	C4—C5—C14—O1	−177.10 (17)
N3—C4—C5—S1	−1.33 (17)	S1—C5—C14—O1	2.19 (19)
C13—C4—C5—S1	175.36 (15)	C4—C5—C14—C15	2.2 (3)
C2—S1—C5—C4	−0.36 (13)	S1—C5—C14—C15	−178.54 (14)
C2—S1—C5—C14	−179.80 (12)	C9—C10—N16—O3	−10.8 (3)
C4—N3—C7—C8	−94.0 (2)	C11—C10—N16—O3	169.3 (2)
C2—N3—C7—C8	85.8 (2)	C9—C10—N16—O2	169.77 (19)
C4—N3—C7—C12	85.2 (2)	C11—C10—N16—O2	−10.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···O1^{i}	0.85 (2)	2.39 (2)	3.174 (2)	153 (2)

C8—H8···O1^{ii}	0.93	2.29	3.209 (2)	168
C15—H15B···O1^{iii}	0.96	2.49	3.434 (2)	166

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

Fig. 1

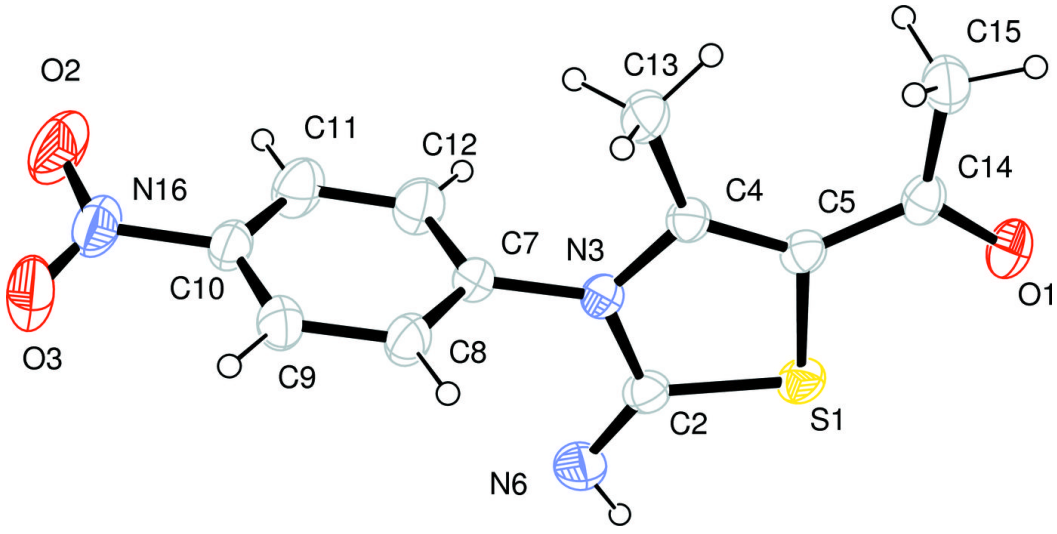


Fig. 2