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Ethyl (*Z*)-2-[2-(4-methylphenyl)hydrazin-1-ylidene]-3-oxo-3-(thiazol-2-ylamino)propanoate

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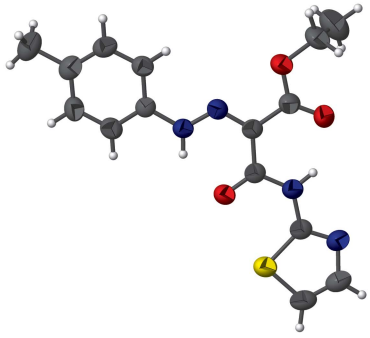
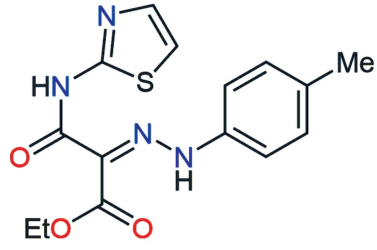
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{16}N_4O_3S$, the dihedral angle between the aromatic rings is 15.90 (19)°. The molecule features two intramolecular N—H···O hydrogen bonds, which both close *S*(6) rings. In the crystal, weak C—H···O interactions link the molecules into [010] chains.

3D view



Chemical scheme



Structure description

Azo dyes containing thiazole ring systems have various industrial applications (*e.g.* Hunger, 2003; El-Shishtawy *et al.*, 2013). As part of our studies in this area, we now describe the synthesis and structure of the title compound (Fig. 1).

The central atoms (C4/C5/C6/O1/O2/O3/N2/N3/N4) are almost coplanar (r.m.s. deviation = 0.037 Å) and subtend dihedral angles of 10.35 (16) and 6.63 (15)° with the five- and six-membered rings, respectively. The dihedral angle between the rings is 15.90 (19)°. The terminal CH_3 group of the ethyl side-chain is twisted away from the rest of the molecule [C6—O3—C7—C8 = 81.4 (5)°]. The molecule features two intramolecular N—H···O hydrogen bonds (Table 1), which both generate *S*(6) rings. In the crystal, weak C—H···O interactions link the molecules into [010] *C*(8) chains (Fig. 2), with adjacent molecules related by the 2_1 screw axis.

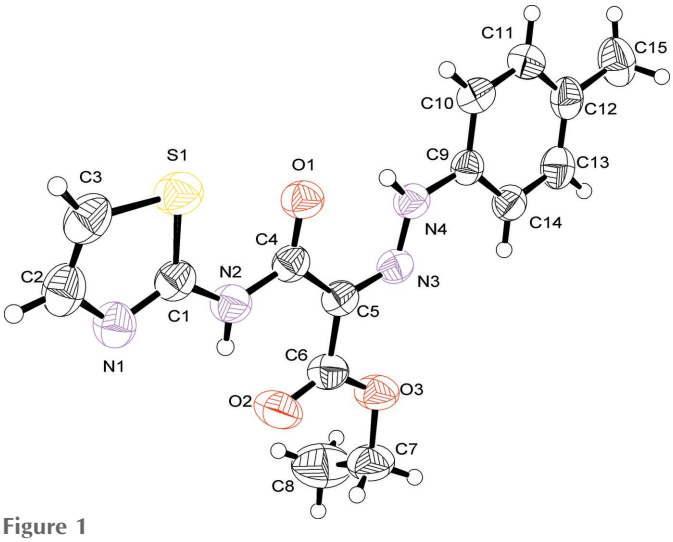


Figure 1
The molecular structure showing 50% displacement ellipsoids.

Synthesis and crystallization

1-Chloro-2-(4-tolyl)diazene and ethyl 3-oxo-3-(thiazol-2-yl-amino)propanoate were dissolved in ethanol containing sodium acetate trihydrate and placed in an ice-bath for 2 h. The resulting solid was filtered, washed with ethanol, dried and recrystallized from dimethylformamide solution to give yellow blocks, m.p. 152–153°C.

Refinement

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

Funding information

The project was supported by King Saud University, Deanship of Scientific Research, Research Chairs.

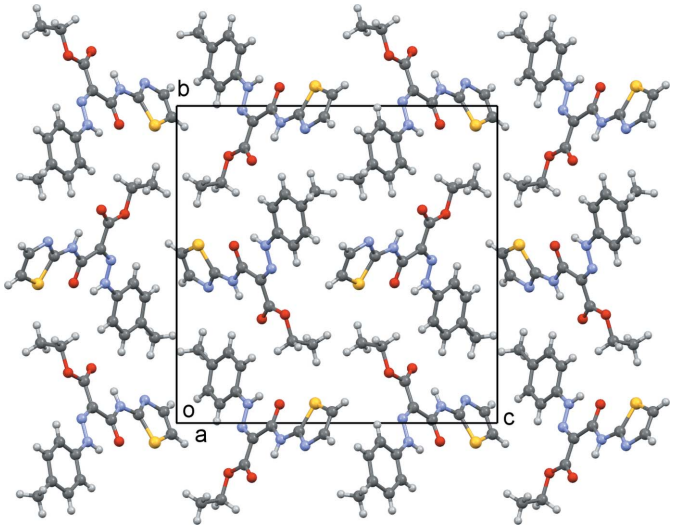


Figure 2
A view down the *a* axis of the crystal packing.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.86	1.93	2.639 (4)	139
N4—H4···O1	0.86	1.91	2.590 (4)	135
C10—H10···O2^{i}	0.93	2.44	3.255 (5)	147

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{16}N_4O_3S$
M_r	332.38
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	298
a, *b*, *c* (Å)	5.6487 (3), 16.7479 (11), 16.9726 (14)
V (Å^3)	1605.67 (19)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.22
Crystal size (mm)	0.18 × 0.18 × 0.11
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent 2014)
T_{min}, T_{max}	0.756, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5888, 3601, 2691
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.698
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.048, 0.130, 1.07
No. of reflections	3601
No. of parameters	210
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.19
Absolute structure	Flack *x* determined using 863 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.02 (5)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 20015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

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full crystallographic data

IUCrData (2018). **3**, x171852 [https://doi.org/10.1107/S2414314617018521]

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Crystal data

$C_{15}H_{16}N_4O_3S$
M_r = 332.38
Orthorhombic, $P2_12_12_1$
a = 5.6487 (3) Å
b = 16.7479 (11) Å
c = 16.9726 (14) Å
V = 1605.67 (19) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.375 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1846 reflections
θ = 3.8–26.8°
μ = 0.22 mm^{-1}
T = 298 K
Block, yellow
0.18 × 0.18 × 0.11 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: multi-scan (CrysAlisPro; Agilent 2014)
T_{min} = 0.756, T_{max} = 1.000
5888 measured reflections
3601 independent reflections
2691 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 29.8°, θ_{min} = 3.4°
h = −7→5
k = −23→15
l = −22→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.130
S = 1.07
3601 reflections
210 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.2718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack x determined using 863 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.02 (5)

Special details

Experimental. Version 1.171.37.35g (release 09-12-2014 CrysAlis171 .NET) (compiled Dec 9 2014,15:38:47) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

Refinement. All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl C—H bonds were fixed at 0.96 Å, with displacement parameters 1.5 times U_{eq}(C), and were allowed to spin about the C—C bonds. The N—H bonds were was fixed at 0.86 Å, ethyl C—H were fixed at 0.97 Å and aromatic C—H distances were set to 0.93 Å and their U(iso) set to 1.2 times the U_{eq} for the atoms to which they are bonded.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5357 (7)	−0.0268 (2)	0.3815 (2)	0.0505 (8)
C2	0.2323 (7)	−0.0349 (3)	0.4602 (2)	0.0648 (11)
H2	0.0977	−0.0579	0.4820	0.078*
C3	0.3136 (8)	0.0352 (3)	0.4845 (3)	0.0663 (11)
H3	0.2458	0.0657	0.5244	0.080*
C4	0.8791 (6)	−0.0096 (2)	0.2963 (2)	0.0466 (8)
C5	1.0331 (6)	−0.0486 (2)	0.2376 (2)	0.0460 (8)
C6	0.9915 (7)	−0.1316 (2)	0.2118 (2)	0.0547 (9)
C7	1.1328 (9)	−0.2416 (2)	0.1390 (3)	0.0760 (13)
H7A	1.0872	−0.2732	0.1844	0.091*
H7B	1.2855	−0.2607	0.1209	0.091*
C8	0.9588 (13)	−0.2523 (3)	0.0768 (4)	0.113 (2)
H8A	0.8081	−0.2325	0.0942	0.170*
H8B	0.9454	−0.3080	0.0642	0.170*
H8C	1.0079	−0.2233	0.0308	0.170*
C9	1.4593 (6)	0.09900 (19)	0.1852 (2)	0.0441 (7)
C10	1.4866 (7)	0.1794 (2)	0.1996 (2)	0.0570 (9)
H10	1.3856	0.2053	0.2346	0.068*
C11	1.6640 (7)	0.2217 (2)	0.1622 (3)	0.0595 (10)
H11	1.6807	0.2761	0.1718	0.071*
C12	1.8173 (6)	0.1838 (2)	0.1106 (2)	0.0576 (10)
C13	1.7886 (7)	0.1027 (2)	0.0987 (2)	0.0582 (10)
H13	1.8923	0.0762	0.0651	0.070*
C14	1.6118 (6)	0.0601 (2)	0.1349 (2)	0.0506 (8)
H14	1.5954	0.0056	0.1256	0.061*
C15	2.0080 (7)	0.2309 (3)	0.0683 (3)	0.0792 (13)
H15A	2.1610	0.2117	0.0840	0.119*
H15B	1.9941	0.2864	0.0817	0.119*
H15C	1.9898	0.2245	0.0124	0.119*
N1	0.3575 (6)	−0.07177 (19)	0.40072 (19)	0.0589 (8)
N2	0.6934 (6)	−0.05218 (19)	0.32443 (18)	0.0545 (7)
H2A	0.6717	−0.0991	0.3051	0.065*
N3	1.2153 (5)	−0.01322 (15)	0.20449 (17)	0.0466 (7)
N4	1.2696 (5)	0.05964 (17)	0.22258 (17)	0.0486 (7)
H4	1.1887	0.0843	0.2579	0.058*
O1	0.9189 (4)	0.05855 (15)	0.32087 (15)	0.0554 (6)

O2	0.8283 (6)	−0.17229 (15)	0.2343 (2)	0.0779 (9)
O3	1.1534 (5)	−0.15850 (15)	0.16202 (17)	0.0660 (8)
S1	0.5615 (2)	0.06154 (6)	0.43218 (7)	0.0657 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0535 (19)	0.0531 (18)	0.0449 (19)	0.0073 (18)	0.0004 (17)	0.0047 (16)
C2	0.057 (2)	0.082 (3)	0.055 (2)	0.009 (2)	0.0076 (19)	0.004 (2)
C3	0.069 (2)	0.073 (3)	0.057 (2)	0.017 (2)	0.012 (2)	−0.001 (2)
C4	0.0492 (19)	0.0473 (18)	0.0432 (19)	0.0053 (15)	−0.0005 (16)	0.0061 (16)
C5	0.0506 (18)	0.0463 (17)	0.0411 (18)	0.0036 (16)	0.0013 (15)	0.0031 (15)
C6	0.065 (2)	0.0471 (17)	0.052 (2)	0.0013 (18)	0.007 (2)	0.0016 (17)
C7	0.092 (3)	0.049 (2)	0.087 (3)	0.004 (2)	0.013 (3)	−0.014 (2)
C8	0.154 (5)	0.078 (3)	0.109 (4)	−0.006 (4)	−0.015 (5)	−0.017 (3)
C9	0.0419 (16)	0.0476 (16)	0.0427 (18)	0.0029 (15)	−0.0039 (15)	0.0034 (15)
C10	0.056 (2)	0.0488 (18)	0.066 (3)	0.0071 (17)	0.001 (2)	−0.0044 (18)
C11	0.056 (2)	0.0474 (19)	0.075 (3)	−0.0039 (17)	−0.002 (2)	0.0031 (19)
C12	0.0449 (18)	0.073 (2)	0.055 (2)	0.0000 (19)	−0.0072 (19)	0.010 (2)
C13	0.048 (2)	0.076 (2)	0.051 (2)	0.0026 (19)	0.0016 (18)	−0.0035 (19)
C14	0.0485 (18)	0.0523 (19)	0.051 (2)	0.0036 (17)	−0.0027 (16)	−0.0048 (18)
C15	0.060 (2)	0.099 (3)	0.078 (3)	−0.015 (2)	−0.001 (2)	0.020 (3)
N1	0.0590 (18)	0.066 (2)	0.0521 (18)	−0.0005 (16)	0.0080 (16)	0.0017 (16)
N2	0.0598 (17)	0.0491 (15)	0.0545 (18)	0.0006 (15)	0.0110 (15)	−0.0029 (14)
N3	0.0504 (16)	0.0443 (14)	0.0452 (16)	0.0040 (13)	−0.0026 (14)	0.0020 (13)
N4	0.0494 (15)	0.0457 (14)	0.0508 (17)	0.0036 (14)	0.0051 (13)	−0.0029 (14)
O1	0.0627 (15)	0.0486 (13)	0.0549 (15)	0.0013 (13)	0.0058 (12)	−0.0047 (12)
O2	0.090 (2)	0.0508 (14)	0.094 (2)	−0.0145 (15)	0.035 (2)	−0.0115 (15)
O3	0.0793 (18)	0.0462 (13)	0.0725 (19)	−0.0003 (13)	0.0211 (16)	−0.0099 (13)
S1	0.0756 (6)	0.0558 (5)	0.0656 (6)	0.0035 (5)	0.0145 (5)	−0.0067 (5)

Geometric parameters (Å, º)

C1—N1	1.299 (5)	C8—H8B	0.9600
C1—N2	1.383 (5)	C8—H8C	0.9600
C1—S1	1.718 (4)	C9—C14	1.377 (5)
C2—C3	1.326 (6)	C9—C10	1.378 (5)
C2—N1	1.378 (5)	C9—N4	1.409 (4)
C2—H2	0.9300	C10—C11	1.381 (5)
C3—S1	1.717 (4)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.385 (6)
C4—O1	1.236 (4)	C11—H11	0.9300
C4—N2	1.354 (4)	C12—C13	1.382 (5)
C4—C5	1.476 (5)	C12—C15	1.516 (5)
C5—N3	1.313 (4)	C13—C14	1.373 (5)
C5—C6	1.475 (5)	C13—H13	0.9300
C6—O2	1.209 (4)	C14—H14	0.9300
C6—O3	1.325 (4)	C15—H15A	0.9600

C7—O3	1.451 (4)	C15—H15B	0.9600
C7—C8	1.454 (7)	C15—H15C	0.9600
C7—H7A	0.9700	N2—H2A	0.8600
C7—H7B	0.9700	N3—N4	1.295 (4)
C8—H8A	0.9600	N4—H4	0.8600

N1—C1—N2	119.8 (3)	C10—C9—N4	117.6 (3)
N1—C1—S1	116.1 (3)	C9—C10—C11	120.0 (4)
N2—C1—S1	124.1 (3)	C9—C10—H10	120.0
C3—C2—N1	116.6 (4)	C11—C10—H10	120.0
C3—C2—H2	121.7	C10—C11—C12	120.7 (4)
N1—C2—H2	121.7	C10—C11—H11	119.7
C2—C3—S1	110.4 (3)	C12—C11—H11	119.7
C2—C3—H3	124.8	C13—C12—C11	117.9 (4)
S1—C3—H3	124.8	C13—C12—C15	121.7 (4)
O1—C4—N2	120.5 (3)	C11—C12—C15	120.4 (4)
O1—C4—C5	121.9 (3)	C14—C13—C12	122.1 (4)
N2—C4—C5	117.5 (3)	C14—C13—H13	119.0
N3—C5—C6	115.0 (3)	C12—C13—H13	119.0
N3—C5—C4	123.5 (3)	C13—C14—C9	119.1 (4)
C6—C5—C4	121.5 (3)	C13—C14—H14	120.5
O2—C6—O3	122.4 (3)	C9—C14—H14	120.5
O2—C6—C5	124.0 (3)	C12—C15—H15A	109.5
O3—C6—C5	113.6 (3)	C12—C15—H15B	109.5
O3—C7—C8	111.5 (4)	H15A—C15—H15B	109.5
O3—C7—H7A	109.3	C12—C15—H15C	109.5
C8—C7—H7A	109.3	H15A—C15—H15C	109.5
O3—C7—H7B	109.3	H15B—C15—H15C	109.5
C8—C7—H7B	109.3	C1—N1—C2	108.8 (3)
H7A—C7—H7B	108.0	C4—N2—C1	125.7 (3)
C7—C8—H8A	109.5	C4—N2—H2A	117.1
C7—C8—H8B	109.5	C1—N2—H2A	117.1
H8A—C8—H8B	109.5	N4—N3—C5	120.6 (3)
C7—C8—H8C	109.5	N3—N4—C9	121.0 (3)
H8A—C8—H8C	109.5	N3—N4—H4	119.5
H8B—C8—H8C	109.5	C9—N4—H4	119.5
C14—C9—C10	120.2 (3)	C6—O3—C7	116.3 (3)
C14—C9—N4	122.2 (3)	C3—S1—C1	88.2 (2)

N1—C2—C3—S1	0.5 (5)	N2—C1—N1—C2	178.0 (3)
O1—C4—C5—N3	−3.2 (5)	S1—C1—N1—C2	−0.6 (4)
N2—C4—C5—N3	178.4 (3)	C3—C2—N1—C1	0.0 (5)
O1—C4—C5—C6	176.7 (3)	O1—C4—N2—C1	−0.9 (5)
N2—C4—C5—C6	−1.7 (5)	C5—C4—N2—C1	177.6 (3)
N3—C5—C6—O2	−177.8 (4)	N1—C1—N2—C4	176.6 (3)
C4—C5—C6—O2	2.3 (6)	S1—C1—N2—C4	−4.9 (5)
N3—C5—C6—O3	3.3 (5)	C6—C5—N3—N4	−179.8 (3)
C4—C5—C6—O3	−176.6 (3)	C4—C5—N3—N4	0.1 (5)

C14—C9—C10—C11	1.5 (5)	C5—N3—N4—C9	−177.6 (3)
N4—C9—C10—C11	−177.5 (3)	C14—C9—N4—N3	−7.5 (5)
C9—C10—C11—C12	−0.6 (6)	C10—C9—N4—N3	171.5 (3)
C10—C11—C12—C13	−0.8 (6)	O2—C6—O3—C7	−3.4 (6)
C10—C11—C12—C15	178.6 (4)	C5—C6—O3—C7	175.5 (3)
C11—C12—C13—C14	1.4 (6)	C8—C7—O3—C6	81.4 (5)
C15—C12—C13—C14	−178.0 (4)	C2—C3—S1—C1	−0.7 (3)
C12—C13—C14—C9	−0.5 (5)	N1—C1—S1—C3	0.8 (3)
C10—C9—C14—C13	−0.9 (5)	N2—C1—S1—C3	−177.8 (3)
N4—C9—C14—C13	178.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.86	1.93	2.639 (4)	139
N4—H4···O1	0.86	1.91	2.590 (4)	135
C10—H10···O2^{i}	0.93	2.44	3.255 (5)	147

Symmetry code: (i) −*x*+2, *y*+1/2, −*z*+1/2.