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(*E*)-*N*′-(2-Thienylmethylidene)-*p*-toluenesulfonohydrazide

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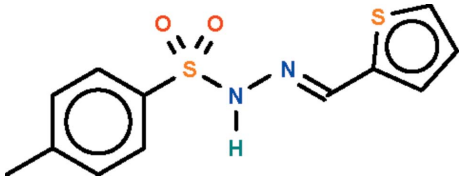
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 18.0.

The S—N(H)—N=C linkage in the title molecule, $C_{12}H_{12}N_2O_2S_2$, is non-planar [torsion angle = 15.5 (1)°] as the amino N atom is pyramidally coordinated. The amino group acts as a hydrogen-bond donor to an O atom of an adjacent molecule, generating chains running parallel to the *c* axis.

Related literature

For the structure of the (*E*)-*N*′-benzylidene-*p*-toluenesulfonohydrazide homolog, see: Mehrabi *et al.* (2008).



Experimental

Crystal data

$C_{12}H_{12}N_2O_2S_2$
M_r = 280.36
Monoclinic, $P2_1/c$
a = 14.3758 (10) Å
b = 9.8613 (7) Å
c = 9.6172 (7) Å
β = 104.981 (1)°
V = 1317.03 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 100 K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.857, T_{max} = 0.925
8238 measured reflections
3022 independent reflections
2728 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.085
S = 1.04
3022 reflections
168 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86 (1)	2.06 (1)	2.874 (2)	159 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank King Abdul Aziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2261).

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supporting information

Acta Cryst. (2010). E**66**, o2360 [https://doi.org/10.1107/S1600536810032708]

(*E*)-*N*′-(2-Thienylmethylidene)-*p*-toluenesulfonohydrazide

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S1. Comment

p-Toluenesulfonyl hydrazide, CH_3-4-$C_6H_4SO_2NHNH_2$, condenses with carbonyl compounds to form Schiff bases, and among the plethora nearly a hundred have had their crystal structures determined. The compounds have the azomethine double-bond in an *E*-configuration. In the Schiff base product between *p*-toluenesulfonyl hydrazide and thiophene-2-carboxaldehyde, the S–N(H)–N═C linkage is non-planar [torsion angle 15.5 (1) °] because the amino nitrogen atom (which bears a hydrogen atom) is pyramidally coordinated (Fig. 1). The amino group acts as a hydrogen-bond donor to an oxygen atom of an adjacent molecule to generate a chain running along the *c*-axis of the monoclinic cell (Fig. 2). The oxygen atom involved in hydrogen bonding [S–O 1.4355 (10) Å] is marginally farther from the sulfur atom than the oxygen that is not involved in hydrogen bonding [S–O 1.4288 (10) Å].

S2. Experimental

p-Toluenesulfonyl hydrazide (4.66 g, 2.5 mmol) and thiophene-2-carboxaldehyde (2.80 g, 2.5 mmol) were heated in methanol (50 ml) for two hours. The cool solution yielded a precipitate that was recrystallized from ethanol and collected in 90% yield.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.99 Å, *U*(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint [N–H 0.86 (1) Å]; its temperature factor was freely refined.

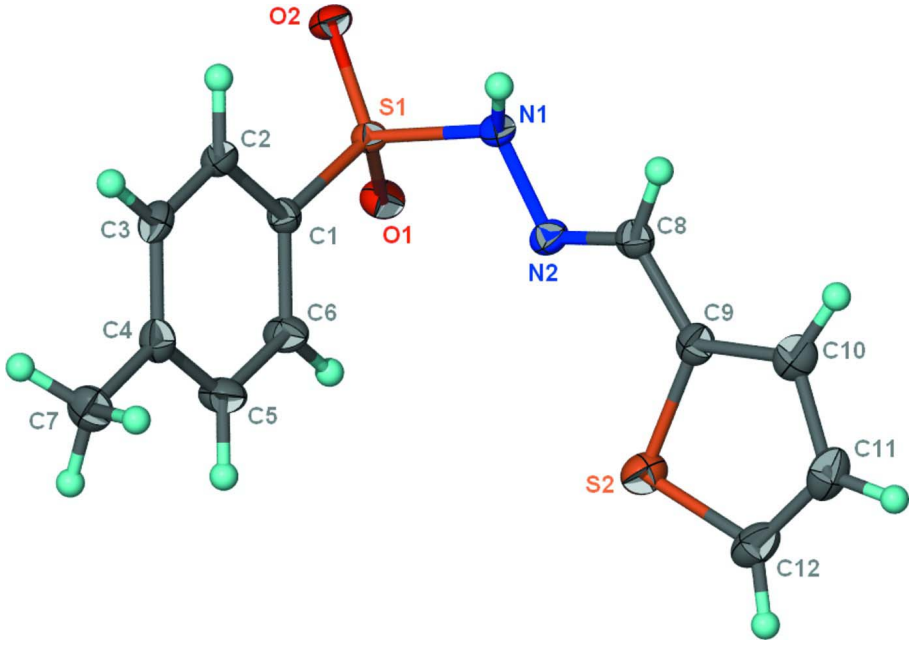

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{12}H_{12}N_2O_2S_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

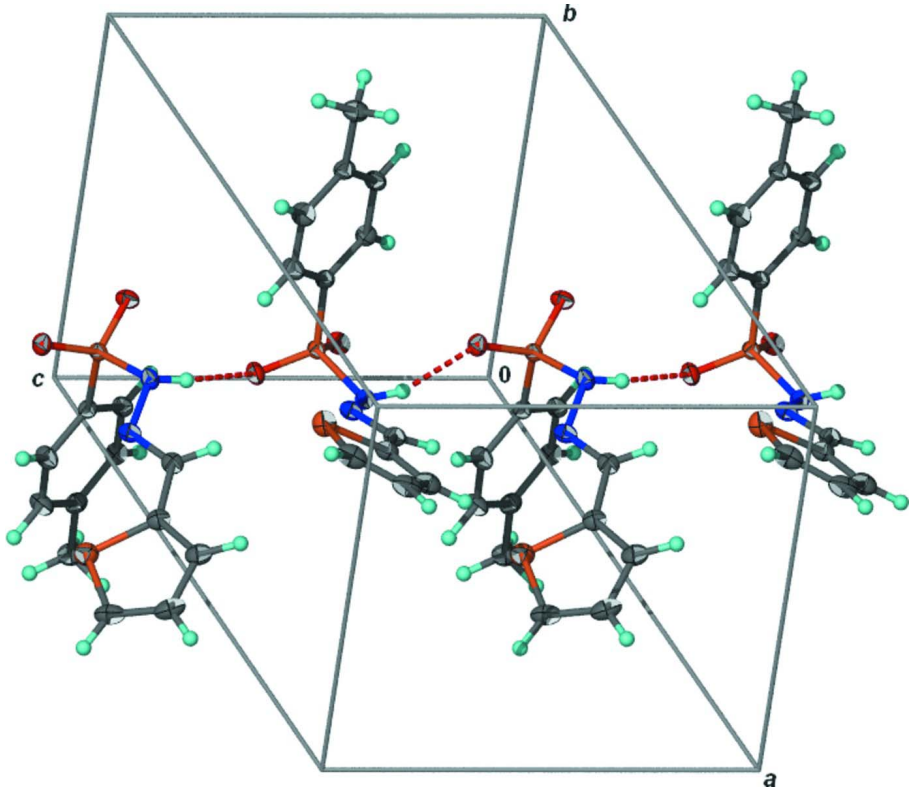

Figure 2

A view of the chain structure resulting from N—H···O hydrogen-bonding.

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Crystal data

$C_{12}H_{12}N_2O_2S_2$
$M_r = 280.36$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.3758\ (10)$ Å
$b = 9.8613\ (7)$ Å
$c = 9.6172\ (7)$ Å
$\beta = 104.981\ (1)°$
$V = 1317.03\ (16)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4750 reflections
$\theta = 2.5–28.3°$
$\mu = 0.40$ mm^{-1}
$T = 100$ K
Prism, yellow
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.857$, $T_{max} = 0.925$
8238 measured reflections
3022 independent reflections
2728 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
$h = -18\rightarrow18$
$k = -12\rightarrow12$
$l = -8\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.085$
$S = 1.04$
3022 reflections
168 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.7746P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.16267 (2)	0.25347 (3)	0.52894 (3)	0.01262 (10)
S2	0.51633 (3)	0.43030 (4)	0.75091 (4)	0.02186 (11)
O1	0.18378 (8)	0.21042 (11)	0.67638 (11)	0.0176 (2)
O2	0.08674 (7)	0.18970 (10)	0.42362 (11)	0.0174 (2)
N1	0.26105 (9)	0.22234 (13)	0.47478 (13)	0.0148 (2)
H1	0.2522 (14)	0.235 (2)	0.3840 (11)	0.024 (5)*
N2	0.34437 (9)	0.28097 (12)	0.56466 (13)	0.0156 (2)
C1	0.14605 (10)	0.42992 (14)	0.52115 (15)	0.0136 (3)
C2	0.08653 (10)	0.48722 (15)	0.39742 (15)	0.0159 (3)
H2	0.0530	0.4313	0.3203	0.019*
C3	0.07698 (10)	0.62763 (15)	0.38847 (15)	0.0170 (3)
H3	0.0360	0.6673	0.3048	0.020*

C4	0.12658 (10)	0.71101 (15)	0.50031 (16)	0.0172 (3)
C5	0.18520 (11)	0.65074 (15)	0.62312 (16)	0.0206 (3)
H5	0.2189	0.7065	0.7003	0.025*
C6	0.19530 (11)	0.51082 (15)	0.63480 (15)	0.0191 (3)
H6	0.2353	0.4710	0.7192	0.023*
C7	0.11737 (12)	0.86307 (15)	0.48857 (17)	0.0216 (3)
H7A	0.1290	0.9025	0.5851	0.032*
H7B	0.0524	0.8870	0.4322	0.032*
H7C	0.1647	0.8987	0.4408	0.032*
C8	0.41651 (11)	0.28677 (14)	0.51016 (16)	0.0169 (3)
H8	0.4109	0.2523	0.4161	0.020*
C9	0.50662 (10)	0.34545 (15)	0.59069 (16)	0.0173 (3)
C10	0.59275 (11)	0.34852 (15)	0.55085 (17)	0.0208 (3)
H10	0.6019	0.3079	0.4658	0.025*
C11	0.66556 (11)	0.41995 (16)	0.65279 (19)	0.0235 (3)
H11	0.7291	0.4324	0.6431	0.028*
C12	0.63483 (11)	0.46843 (16)	0.76529 (18)	0.0238 (3)
H12	0.6745	0.5179	0.8432	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01349 (18)	0.01241 (17)	0.01124 (17)	−0.00057 (12)	0.00189 (13)	0.00002 (11)
S2	0.0198 (2)	0.0240 (2)	0.0209 (2)	−0.00308 (14)	0.00374 (15)	−0.00061 (14)
O1	0.0225 (5)	0.0173 (5)	0.0128 (5)	−0.0004 (4)	0.0045 (4)	0.0019 (4)
O2	0.0160 (5)	0.0164 (5)	0.0175 (5)	−0.0029 (4)	0.0002 (4)	−0.0010 (4)
N1	0.0137 (6)	0.0176 (6)	0.0120 (6)	0.0007 (4)	0.0014 (4)	−0.0019 (4)
N2	0.0142 (6)	0.0142 (5)	0.0163 (6)	−0.0001 (4)	0.0000 (5)	0.0006 (4)
C1	0.0136 (6)	0.0128 (6)	0.0149 (6)	0.0002 (5)	0.0048 (5)	0.0003 (5)
C2	0.0133 (6)	0.0180 (7)	0.0152 (6)	−0.0005 (5)	0.0018 (5)	−0.0011 (5)
C3	0.0136 (6)	0.0192 (7)	0.0168 (7)	0.0024 (5)	0.0017 (5)	0.0031 (5)
C4	0.0161 (7)	0.0159 (7)	0.0210 (7)	0.0011 (5)	0.0073 (6)	0.0017 (5)
C5	0.0249 (7)	0.0170 (7)	0.0177 (7)	−0.0016 (6)	0.0013 (6)	−0.0032 (6)
C6	0.0226 (7)	0.0179 (7)	0.0138 (7)	0.0015 (6)	−0.0005 (5)	0.0002 (5)
C7	0.0246 (8)	0.0143 (7)	0.0256 (8)	0.0008 (6)	0.0062 (6)	0.0017 (6)
C8	0.0184 (7)	0.0140 (6)	0.0176 (7)	0.0017 (5)	0.0032 (5)	0.0006 (5)
C9	0.0169 (7)	0.0145 (6)	0.0199 (7)	0.0012 (5)	0.0038 (5)	0.0026 (5)
C10	0.0203 (7)	0.0163 (7)	0.0248 (8)	−0.0010 (6)	0.0040 (6)	0.0040 (6)
C11	0.0164 (7)	0.0190 (7)	0.0340 (9)	−0.0011 (6)	0.0048 (6)	0.0077 (6)
C12	0.0189 (7)	0.0192 (7)	0.0293 (8)	−0.0042 (6)	−0.0006 (6)	0.0035 (6)

Geometric parameters (Å, º)

S1—O2	1.4288 (10)	C4—C7	1.507 (2)
S1—O1	1.4355 (10)	C5—C6	1.389 (2)
S1—N1	1.6572 (13)	C5—H5	0.9500
S1—C1	1.7553 (14)	C6—H6	0.9500
S2—C12	1.7148 (16)	C7—H7A	0.9800

S2—C9	1.7270 (15)	C7—H7B	0.9800
N1—N2	1.4074 (16)	C7—H7C	0.9800
N1—H1	0.859 (9)	C8—C9	1.447 (2)
N2—C8	1.279 (2)	C8—H8	0.9500
C1—C6	1.3901 (19)	C9—C10	1.388 (2)
C1—C2	1.3932 (19)	C10—C11	1.422 (2)
C2—C3	1.392 (2)	C10—H10	0.9500
C2—H2	0.9500	C11—C12	1.357 (2)
C3—C4	1.395 (2)	C11—H11	0.9500
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.394 (2)		

O2—S1—O1	119.85 (6)	C4—C5—H5	119.3
O2—S1—N1	104.68 (6)	C1—C6—C5	119.03 (13)
O1—S1—N1	106.02 (6)	C1—C6—H6	120.5
O2—S1—C1	109.60 (6)	C5—C6—H6	120.5
O1—S1—C1	109.08 (7)	C4—C7—H7A	109.5
N1—S1—C1	106.74 (6)	C4—C7—H7B	109.5
C12—S2—C9	91.59 (8)	H7A—C7—H7B	109.5
N2—N1—S1	113.01 (9)	C4—C7—H7C	109.5
N2—N1—H1	116.3 (13)	H7A—C7—H7C	109.5
S1—N1—H1	112.1 (13)	H7B—C7—H7C	109.5
C8—N2—N1	114.73 (12)	N2—C8—C9	120.48 (14)
C6—C1—C2	120.95 (13)	N2—C8—H8	119.8
C6—C1—S1	119.95 (11)	C9—C8—H8	119.8
C2—C1—S1	119.03 (11)	C10—C9—C8	126.88 (14)
C1—C2—C3	119.03 (13)	C10—C9—S2	111.33 (11)
C1—C2—H2	120.5	C8—C9—S2	121.73 (11)
C3—C2—H2	120.5	C9—C10—C11	111.76 (14)
C4—C3—C2	121.08 (13)	C9—C10—H10	124.1
C4—C3—H3	119.5	C11—C10—H10	124.1
C2—C3—H3	119.5	C12—C11—C10	113.01 (14)
C3—C4—C5	118.60 (13)	C12—C11—H11	123.5
C3—C4—C7	120.76 (13)	C10—C11—H11	123.5
C5—C4—C7	120.65 (14)	C11—C12—S2	112.32 (12)
C6—C5—C4	121.31 (14)	C11—C12—H12	123.8
C6—C5—H5	119.3	S2—C12—H12	123.8

O2—S1—N1—N2	178.54 (9)	C3—C4—C5—C6	0.5 (2)
O1—S1—N1—N2	−53.83 (11)	C7—C4—C5—C6	−179.24 (15)
C1—S1—N1—N2	62.38 (11)	C2—C1—C6—C5	−0.6 (2)
S1—N1—N2—C8	−164.50 (11)	S1—C1—C6—C5	176.50 (12)
O2—S1—C1—C6	163.60 (12)	C4—C5—C6—C1	0.2 (2)
O1—S1—C1—C6	30.58 (14)	N1—N2—C8—C9	179.61 (12)
N1—S1—C1—C6	−83.57 (13)	N2—C8—C9—C10	174.24 (14)
O2—S1—C1—C2	−19.24 (14)	N2—C8—C9—S2	−9.0 (2)
O1—S1—C1—C2	−152.26 (11)	C12—S2—C9—C10	−0.45 (12)
N1—S1—C1—C2	93.59 (12)	C12—S2—C9—C8	−177.63 (13)

C6—C1—C2—C3	0.2 (2)	C8—C9—C10—C11	177.28 (14)
S1—C1—C2—C3	−176.95 (11)	S2—C9—C10—C11	0.27 (16)
C1—C2—C3—C4	0.6 (2)	C9—C10—C11—C12	0.12 (19)
C2—C3—C4—C5	−1.0 (2)	C10—C11—C12—S2	−0.46 (18)
C2—C3—C4—C7	178.80 (14)	C9—S2—C12—C11	0.52 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86 (1)	2.06 (1)	2.874 (2)	159 (2)

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