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(E)-5-Phenyl-N-(2-thienylmethylene)-1,3,4-thiadiazole-2-amineGüneş Demirtaş,^a Necmi Dege,^a Memet Şekerci,^b Süleyman Servi^b and Muharrem Dinçer^{a*}^aDepartment of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey, and ^bDepartment of Chemistry, Faculty of Arts and Sciences, Firat University, 38039 Elazığ, Turkey

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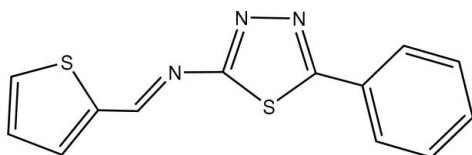
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.026; wR factor = 0.080; data-to-parameter ratio = 14.9.

In the title compound, $\text{C}_{13}\text{H}_9\text{N}_3\text{S}_2$, the thiophene and phenyl rings are oriented at dihedral angles of 8.00 (7) and 6.31 (7)°, respectively, with respect to the central thiadiazole ring. No significant $\text{C}-\text{H}\cdots\text{S}$ and $\pi-\pi$ interactions exist in the crystal structure.

Related literature

For the biological activity of [1,3,4]-thiadiazole-containing compounds, see: Foroumadi, Soltani *et al.* (2003); Foroumadi, Mansouri *et al.* (2003); Holla *et al.* (2002); Genc & Servi (2005); Servi *et al.* (2005). For a related structure, see: Dege *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_9\text{N}_3\text{S}_2$ $M_r = 271.35$

Monoclinic, $P2_1/c$
 $a = 6.2238$ (3) Å
 $b = 7.7393$ (3) Å
 $c = 25.6959$ (13) Å
 $\beta = 94.701$ (4)°
 $V = 1233.55$ (10) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.41$ mm⁻¹
 $T = 293$ K
 $0.74 \times 0.48 \times 0.16$ mm

Data collection

Stoe IPDS-2 diffractometer
 Absorption correction: integration
 (*X-RED*; Stoe & Cie, 2002)
 $T_{\min} = 0.815$, $T_{\max} = 0.943$

10930 measured reflections
 2619 independent reflections
 2275 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.080$
 $S = 1.05$
 2619 reflections
 176 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.18$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Data collection: *X-Area* (Stoe & Cie, 2002); cell refinement: *X-Area*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2829).

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supporting information

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(E)-5-Phenyl-N-(2-thienylmethylene)-1,3,4-thiadiazole-2-amine

Güneş Demirtaş, Necmi Dege, Memet Şekerci, Süleyman Servi and Muharrem Dinçer

S1. Comment

[1,3,4]-Thiadiazoles and their derivatives exhibit diverse biological activities possibly due to the presence of =N—C—S moiety (Holla *et al.*, 2002; Servi *et al.*, 2005; Genc & Servi, 2005). Various phenyl substituted [1,3,4]-thiazole-2-amines and their derivatives have recently received significant importance because of their diverse biological properties (Foroumadi, Soltani *et al.*, 2003; Foroumadi, Mansouri *et al.*, 2003). We report here the crystal structure of the title compound, (I).

In (I), the C7—S1 [1.7234 (13) Å] distance is shorter than the C8—S1 distance [1.7411 (13) Å]. The C11—S2 distance of 1.6993 (17) Å is shorter than the C10—S2 distance of 1.7229 (13) Å and other C—S bonds in the molecule. These bond distances agree well with the corresponding values reported for 1-(thiophen-2-ylmethyl)-2-(thiophen-2-yl)-1*H*-benzimidazole (Dege *et al.*, 2006). The thiophene and phenyl rings are oriented at dihedral angles of 8.00 (7)° and 6.31 (7)°, respectively, with respect to the central thiadiazole ring.

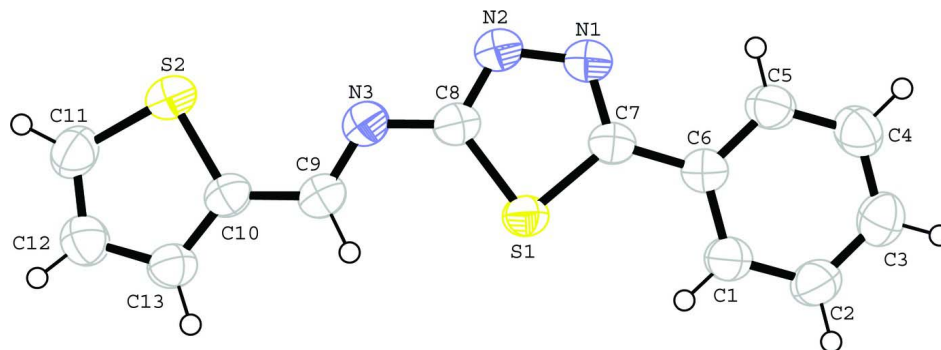
No significant C—H⋯S and π - π interactions are observed.

S2. Experimental

A solution of 5-phenyl-1,3,4-thiadiazole-2-amine (0.01 mol) in absolute ethanol (20 ml) was added in small portions to a solution of thiophen-2-carbaldehyde (0.01 mol) in absolute ethanol (30 ml). The reaction mixture was maintained at 343 K for 4 h, cooled and then added to ice-cold water. The resulting solid was washed with water, dried and recrystallized from ethanol (yield 77%). IR (cm⁻¹): 3078 (Ar H), 1633 (C=C), 1589 (C=N); ¹H-NMR: 7.2–7.9 (m, 8H, aromatic protons), 9.2 (s, ¹H, N=CH proton).

S3. Refinement

Atoms H1, H9 and H12 were located in a difference map and refined freely. The other H atoms were positioned geometrically and refined using a riding model, with C-H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(E)-5-Phenyl-N-(2-thienylmethylene)-1,3,4-thiadiazole-2-amine

Crystal data

$C_{13}H_9N_3S_2$

$M_r = 271.35$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.2238 (3) \text{ \AA}$

$b = 7.7393 (3) \text{ \AA}$

$c = 25.6959 (13) \text{ \AA}$

$\beta = 94.701 (4)^\circ$

$V = 1233.55 (10) \text{ \AA}^3$

$Z = 4$

$F(000) = 560$

$D_x = 1.461 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 17067 reflections

$\theta = 1.6\text{--}26.8^\circ$

$\mu = 0.41 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Plate, yellow

$0.74 \times 0.48 \times 0.16 \text{ mm}$

Data collection

Stoe IPDS-2

diffractometer

Radiation source: fine-focus sealed tube

Plane graphite monochromator

Detector resolution: $6.67 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: integration

(*X-RED*; Stoe & Cie, 2002)

$T_{\min} = 0.815$, $T_{\max} = 0.943$

10930 measured reflections

2619 independent reflections

2275 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 26.8^\circ$, $\theta_{\min} = 1.6^\circ$

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -32 \rightarrow 32$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.080$

$S = 1.05$

2619 reflections

176 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

$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.1148P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.18 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.002634 (11)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0290 (2)	0.1603 (2)	0.57838 (6)	0.0543 (3)
C2	0.0105 (3)	0.0893 (2)	0.62710 (6)	0.0615 (4)
H2	0.1311	0.0817	0.6509	0.074*
C3	-0.1859 (3)	0.0297 (2)	0.64059 (6)	0.0621 (4)
H3	-0.1976	-0.0190	0.6733	0.075*
C4	-0.3643 (2)	0.0422 (2)	0.60563 (6)	0.0627 (4)
H4	-0.4967	0.0014	0.6147	0.075*
C5	-0.3479 (2)	0.1152 (2)	0.55709 (6)	0.0565 (3)
H5	-0.4698	0.1247	0.5338	0.068*
C6	-0.1503 (2)	0.17452 (16)	0.54281 (5)	0.0461 (3)
C7	-0.13539 (19)	0.24791 (17)	0.49053 (5)	0.0466 (3)
C8	-0.0473 (2)	0.36506 (18)	0.40911 (5)	0.0500 (3)
C9	0.2276 (2)	0.46866 (18)	0.36247 (5)	0.0500 (3)
C10	0.3030 (2)	0.55163 (17)	0.31753 (5)	0.0480 (3)
C11	0.3279 (3)	0.7018 (2)	0.23422 (6)	0.0643 (4)
H11	0.3078	0.7541	0.2016	0.077*
C12	0.5205 (3)	0.6920 (2)	0.26256 (7)	0.0631 (4)
C13	0.5080 (2)	0.60554 (18)	0.31021 (6)	0.0547 (3)
H13	0.6259	0.5869	0.3342	0.066*
N1	-0.30366 (19)	0.27346 (19)	0.45777 (5)	0.0656 (4)
N2	-0.25288 (19)	0.3405 (2)	0.41099 (5)	0.0674 (4)
N3	0.02776 (18)	0.43483 (15)	0.36467 (4)	0.0530 (3)
S1	0.10458 (5)	0.30465 (5)	0.466131 (13)	0.04910 (12)
S2	0.12673 (6)	0.60757 (5)	0.265024 (14)	0.06061 (14)
H9	0.333 (3)	0.444 (2)	0.3905 (6)	0.065 (4)*
H1	0.158 (3)	0.206 (2)	0.5694 (7)	0.071 (5)*
H12	0.645 (3)	0.736 (3)	0.2497 (8)	0.086 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0472 (7)	0.0616 (8)	0.0537 (7)	-0.0008 (6)	0.0013 (6)	-0.0007 (6)
C2	0.0579 (8)	0.0733 (10)	0.0520 (8)	0.0033 (7)	-0.0038 (6)	0.0016 (7)
C3	0.0714 (9)	0.0656 (9)	0.0502 (8)	0.0040 (7)	0.0103 (7)	0.0006 (7)
C4	0.0557 (8)	0.0725 (10)	0.0617 (9)	-0.0027 (7)	0.0151 (7)	-0.0015 (7)
C5	0.0455 (7)	0.0690 (9)	0.0549 (8)	0.0005 (6)	0.0033 (6)	-0.0047 (7)

C6	0.0460 (6)	0.0435 (7)	0.0486 (6)	0.0036 (5)	0.0032 (5)	-0.0061 (5)
C7	0.0425 (6)	0.0452 (6)	0.0516 (7)	0.0016 (5)	0.0007 (5)	-0.0035 (5)
C8	0.0466 (7)	0.0514 (7)	0.0509 (7)	0.0011 (5)	-0.0026 (5)	0.0001 (6)
C9	0.0513 (7)	0.0494 (7)	0.0483 (7)	0.0046 (6)	-0.0021 (5)	-0.0003 (6)
C10	0.0496 (7)	0.0469 (7)	0.0467 (7)	0.0051 (5)	-0.0010 (5)	-0.0019 (5)
C11	0.0729 (10)	0.0655 (9)	0.0537 (8)	-0.0013 (7)	0.0004 (7)	0.0108 (7)
C12	0.0590 (8)	0.0636 (10)	0.0670 (9)	-0.0037 (7)	0.0073 (7)	0.0085 (7)
C13	0.0503 (7)	0.0552 (8)	0.0576 (8)	0.0031 (6)	-0.0020 (6)	0.0031 (6)
N1	0.0443 (6)	0.0864 (9)	0.0647 (8)	-0.0049 (6)	-0.0039 (5)	0.0167 (7)
N2	0.0479 (6)	0.0901 (10)	0.0622 (7)	-0.0051 (6)	-0.0068 (5)	0.0208 (7)
N3	0.0518 (6)	0.0564 (7)	0.0499 (6)	-0.0003 (5)	-0.0009 (5)	0.0045 (5)
S1	0.04071 (18)	0.0581 (2)	0.04783 (19)	0.00331 (13)	-0.00016 (12)	0.00054 (14)
S2	0.0538 (2)	0.0724 (3)	0.0536 (2)	-0.00062 (16)	-0.00781 (15)	0.00514 (17)

Geometric parameters (Å, °)

C1—C2	1.381 (2)	C8—N3	1.3787 (18)
C1—C6	1.3875 (19)	C8—S1	1.7411 (13)
C1—H1	0.923 (18)	C9—N3	1.2770 (18)
C2—C3	1.377 (2)	C9—C10	1.4335 (19)
C2—H2	0.93	C9—H9	0.952 (16)
C3—C4	1.374 (2)	C10—C13	1.3697 (19)
C3—H3	0.93	C10—S2	1.7229 (13)
C4—C5	1.380 (2)	C11—C12	1.353 (2)
C4—H4	0.93	C11—S2	1.6993 (17)
C5—C6	1.3900 (19)	C11—H11	0.93
C5—H5	0.93	C12—C13	1.403 (2)
C6—C7	1.4684 (19)	C12—H12	0.929 (19)
C7—N1	1.3039 (17)	C13—H13	0.93
C7—S1	1.7234 (13)	N1—N2	1.3696 (19)
C8—N2	1.2987 (18)		
C2—C1—C6	120.44 (14)	N2—C8—S1	113.44 (11)
C2—C1—H1	121.1 (12)	N3—C8—S1	127.22 (10)
C6—C1—H1	118.3 (11)	N3—C9—C10	120.78 (12)
C3—C2—C1	120.20 (14)	N3—C9—H9	122.7 (10)
C3—C2—H2	119.9	C10—C9—H9	116.5 (10)
C1—C2—H2	119.9	C13—C10—C9	128.09 (12)
C4—C3—C2	119.90 (14)	C13—C10—S2	110.88 (10)
C4—C3—H3	120.0	C9—C10—S2	120.94 (10)
C2—C3—H3	120.0	C12—C11—S2	112.30 (12)
C3—C4—C5	120.28 (14)	C12—C11—H11	123.9
C3—C4—H4	119.9	S2—C11—H11	123.9
C5—C4—H4	119.9	C11—C12—C13	112.73 (14)
C4—C5—C6	120.40 (14)	C11—C12—H12	121.0 (13)
C4—C5—H5	119.8	C13—C12—H12	126.3 (13)
C6—C5—H5	119.8	C10—C13—C12	112.57 (13)
C1—C6—C5	118.77 (13)	C10—C13—H13	123.7

C1—C6—C7	121.70 (12)	C12—C13—H13	123.7
C5—C6—C7	119.53 (12)	C7—N1—N2	113.15 (12)
N1—C7—C6	122.79 (12)	C8—N2—N1	112.78 (12)
N1—C7—S1	113.62 (11)	C9—N3—C8	120.94 (12)
C6—C7—S1	123.58 (9)	C7—S1—C8	87.01 (6)
N2—C8—N3	119.33 (12)	C11—S2—C10	91.51 (7)
C6—C1—C2—C3	0.9 (2)	C11—C12—C13—C10	-0.5 (2)
C1—C2—C3—C4	-0.5 (2)	C6—C7—N1—N2	-179.04 (13)
C2—C3—C4—C5	-0.3 (2)	S1—C7—N1—N2	-0.15 (18)
C3—C4—C5—C6	0.9 (2)	N3—C8—N2—N1	-178.89 (13)
C2—C1—C6—C5	-0.3 (2)	S1—C8—N2—N1	0.45 (19)
C2—C1—C6—C7	-179.40 (13)	C7—N1—N2—C8	-0.2 (2)
C4—C5—C6—C1	-0.6 (2)	C10—C9—N3—C8	-175.86 (12)
C4—C5—C6—C7	178.55 (14)	N2—C8—N3—C9	176.50 (15)
C1—C6—C7—N1	-175.17 (14)	S1—C8—N3—C9	-2.7 (2)
C5—C6—C7—N1	5.7 (2)	N1—C7—S1—C8	0.32 (12)
C1—C6—C7—S1	6.05 (19)	C6—C7—S1—C8	179.20 (12)
C5—C6—C7—S1	-173.02 (11)	N2—C8—S1—C7	-0.44 (13)
N3—C9—C10—C13	175.89 (14)	N3—C8—S1—C7	178.84 (13)
N3—C9—C10—S2	-0.24 (19)	C12—C11—S2—C10	-0.24 (14)
S2—C11—C12—C13	0.5 (2)	C13—C10—S2—C11	-0.04 (12)
C9—C10—C13—C12	-176.14 (14)	C9—C10—S2—C11	176.70 (12)
S2—C10—C13—C12	0.30 (17)		
