

**(Z)-2-Methoxy-N-[(5-nitrothiophen-2-yl)-methylidene]aniline**Nihal Kan Kaynar,<sup>a\*</sup> Sümeyye Gümüş,<sup>b</sup> Erbil Ağar,<sup>b</sup> Orhan Büyükgüngör<sup>a</sup> and Metin Yavuz<sup>c</sup><sup>a</sup>Department of Physics, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit-Samsun, Turkey, <sup>b</sup>Department of Chemistry, Faculty of Arts & Science, Ondokuz Mayıs University, 55139 Samsun, Turkey, and <sup>c</sup>Department of Physics, Faculty of Arts & Science, Ondokuz Mayıs University, TR-55139 Kurupelit-Samsun, Turkey, and, Faculty of Technology, Amasya University, TR-05100 Amasya, Turkey

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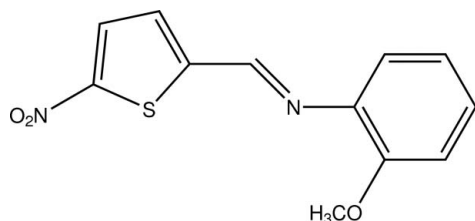
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.069; data-to-parameter ratio = 15.9.

The dihedral angle between the benzene and thiophene rings in the title compound,  $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3\text{S}$ , is  $27.94(13)^\circ$ . An intermolecular  $\text{C}-\text{H}\cdots\pi$  interaction contributes to the stability of the crystal structure.

**Related literature**

For the biological properties of Schiff bases, see: Barton & Ollis (1979); Layer (1963); Ingold (1969), for their industrial properties, see: Taggi *et al.* (2002) and for their reaction properties, see: Aydoğan *et al.* (2001). For related structures, see: Ağar *et al.* (2010); Tanak *et al.* (2009); Ceylan *et al.* (2011).

**Experimental***Crystal data* $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3\text{S}$  $M_r = 262.28$ Orthorhombic,  $P2_12_12_1$  $a = 6.6825(6)$  Å $b = 7.7926(5)$  Å $c = 23.7180(12)$  Å $V = 1235.09(15)$  Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.26$  mm<sup>-1</sup> $T = 296$  K $0.59 \times 0.39 \times 0.05$  mm*Data collection*

Stoe IPDS II diffractometer

Absorption correction: integration  
(*X-RED32*; Stoe & Cie, 2002) $T_{\min} = 0.974$ ,  $T_{\max} = 0.974$ 

5645 measured reflections

2599 independent reflections

1799 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.038$ *Refinement* $R[F^2 > 2\sigma(F^2)] = 0.037$  $wR(F^2) = 0.069$  $S = 0.93$ 

2599 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.12$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

1067 Friedel pairs

Flack parameter:  $-0.04(8)$ **Table 1**

Hydrogen-bond geometry (Å, °).

Cg2 is the centroid of the C6–C11 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10–H10 $\cdots$ Cg2 <sup>i</sup>	0.93	2.77	3.605 (3)	149

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5776).

**References**

- Ağar, A., Tanak, H. & Yavuz, M. (2010). *Mol. Phys.* **108**, 1759–1772.  
 Aydoğan, F., Öcal, N., Turgut, Z. & Yolaçan, C. (2001). *Bull. Korean Chem. Soc.* **22**, 476–480.  
 Barton, D. & Ollis, W. D. (1979). *Comprehensive Organic Chemistry*, Vol. 2. Oxford: Pergamon.  
 Ceylan, Ü., Tanak, H., Gümüş, S. & Ağar, E. (2011). *Acta Cryst.* **E67**, o2004.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Ingold, C. K. (1969). *Structure and Mechanism in Organic Chemistry*, 2nd ed. Ithaca: Cornell University Press.  
 Layer, R. W. (1963). *Chem. Rev.* **63**, 489–510.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.  
 Taggi, A. E., Hafez, A. M., Wack, H., Young, B., Ferraris, D. & Lectka, T. (2002). *J. Am. Chem. Soc.* **124**, 6626–6635.  
 Tanak, H., Erşahin, F., Ağar, E., Yavuz, M. & Büyükgüngör, O. (2009). *Acta Cryst.* **E65**, o2291.

**supplementary materials**

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## (Z)-2-Methoxy-N-[(5-nitrothiophen-2-yl)methylidene]aniline

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### Comment

Schiff bases, *i.e.*, compounds having a double C=N bond, are used as starting materials in the synthesis of important drugs, such as antibiotics and antiallergic, antiphlogistic, and antitumor substances (Barton *et al.*, 1979; Layer, 1963; Ingold 1969). On the industrial scale, they have a wide range of applications, such as dyes and pigments (Taggi *et al.*, 2002). Schiff bases have also been employed as ligands for the complexation of metal ions (Aydoğan *et al.*, 2001).

The molecular structure of the title compound is shown on Fig. 1. The dihedral angle between the C10—C13/S1 nitrothiophene and the C1—C6 phenyl ring is 27.94 (13)°. The deviation from planarity may be due to steric repulsion between the methylene group and phenyl ring. The length of the C5=N2 double bond is 1.266 (3) Å, slightly shorter than standard 1.28 Å value of a C=N double bond and consistent with related structures (Ağar *et al.*, 2010; Tanak *et al.*, 2009; Ceylan *et al.*, 2011).

The crystal structure is stabilized by an intermolecular C—H... $\pi$  interaction (C10—H10...Cg2). No significant  $\pi$ — $\pi$  interactions are observed in the crystal structure.

### Experimental

The compound (Z)—N-(2-methoxyphenyl)-1-(5-nitrothiophen-2-yl)methanimine was prepared by reflux a mixture of a solution containing 5-nitro-2-thiophene-carboxaldehyde (0.0078 g 0.050 mmol) in 20 ml ethanol and a solution containing *o*-Anisidine (0.0062 g 0.050 mmol) in 20 ml ethanol. The reaction mixture was stirred for 1 h under reflux. The crystals of (Z)—N-(2-methoxyphenyl)-1-(5-nitrothiophen-2-yl)methanimine suitable for X-ray analysis were obtained from ethylalcohol by slow evaporation (yield % 76; 85–87 °C).

### Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$ .

### Figures

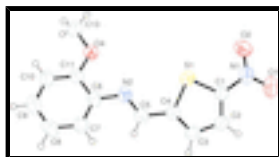


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids.

## (Z)-2-Methoxy-N-[(5-nitrothiophen-2-yl)methylidene]aniline

### Crystal data

$C_{12}H_{10}N_2O_3S$	$F(000) = 544$
$M_r = 262.28$	$D_x = 1.411 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2ac 2ab	Cell parameters from 6785 reflections
$a = 6.6825 (6) \text{ \AA}$	$\theta = 2.6\text{--}27.2^\circ$
$b = 7.7926 (5) \text{ \AA}$	$\mu = 0.26 \text{ mm}^{-1}$
$c = 23.7180 (12) \text{ \AA}$	$T = 296 \text{ K}$
$V = 1235.09 (15) \text{ \AA}^3$	Plate, yellow
$Z = 4$	$0.59 \times 0.39 \times 0.05 \text{ mm}$

### Data collection

Stoe IPDS II diffractometer	2599 independent reflections
Radiation source: fine-focus sealed tube graphite	1799 reflections with $I > 2\sigma(I)$
Detector resolution: $6.67 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.038$
rotation method scans	$\theta_{\text{max}} = 26.8^\circ$ , $\theta_{\text{min}} = 2.8^\circ$
Absorption correction: integration ( <i>X-RED32</i> ; Stoe & Cie, 2002)	$h = -6 \rightarrow 8$
$T_{\text{min}} = 0.974$ , $T_{\text{max}} = 0.974$	$k = -9 \rightarrow 9$
5645 measured reflections	$l = -29 \rightarrow 29$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.069$	$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2]$
$S = 0.93$	where $P = (F_o^2 + 2F_c^2)/3$
2599 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
163 parameters	$\Delta\rho_{\text{max}} = 0.12 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1067 Friedel pairs
	Flack parameter: $-0.04 (8)$

### Special details

**Experimental.** 108 frames, detector distance = 120 mm

**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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.93052 (10)	0.80791 (7)	0.16132 (3)	0.06181 (17)
O4	0.4298 (3)	0.59180 (17)	0.04012 (7)	0.0711 (5)
C7	0.5822 (4)	0.2075 (3)	0.11420 (10)	0.0649 (6)
H7	0.6780	0.1749	0.1405	0.078*
C11	0.4265 (4)	0.4242 (3)	0.05670 (10)	0.0572 (5)
C6	0.5750 (4)	0.3765 (3)	0.09543 (9)	0.0568 (6)
N2	0.7021 (4)	0.5066 (2)	0.11656 (8)	0.0613 (5)
C1	1.1502 (4)	0.8578 (3)	0.19332 (10)	0.0561 (6)
O1	1.3451 (3)	1.0633 (2)	0.23403 (9)	0.0968 (7)
C8	0.4476 (5)	0.0878 (3)	0.09384 (11)	0.0735 (7)
H8	0.4546	-0.0255	0.1060	0.088*
N1	1.1877 (4)	1.0325 (3)	0.20966 (9)	0.0695 (6)
C5	0.8791 (4)	0.4701 (3)	0.13135 (10)	0.0614 (6)
H5	0.9268	0.3592	0.1259	0.074*
C9	0.3052 (5)	0.1357 (3)	0.05614 (12)	0.0744 (8)
H9	0.2147	0.0546	0.0428	0.089*
C4	1.0087 (3)	0.5975 (3)	0.15657 (11)	0.0562 (6)
O2	1.0599 (4)	1.1402 (2)	0.19817 (8)	0.0883 (6)
C10	0.2928 (4)	0.3033 (3)	0.03729 (10)	0.0671 (6)
H10	0.1944	0.3343	0.0115	0.081*
C3	1.1934 (4)	0.5742 (3)	0.17897 (11)	0.0675 (7)
H3	1.2582	0.4686	0.1798	0.081*
C2	1.2765 (4)	0.7248 (3)	0.20070 (11)	0.0685 (7)
H2	1.4013	0.7323	0.2178	0.082*
C13	0.2741 (5)	0.6489 (3)	0.00482 (13)	0.0897 (9)
H13A	0.2933	0.7682	-0.0037	0.135*
H13B	0.1481	0.6339	0.0236	0.135*
H13C	0.2748	0.5837	-0.0295	0.135*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0602 (3)	0.0558 (3)	0.0694 (4)	0.0056 (3)	-0.0122 (4)	0.0013 (3)
O4	0.0705 (11)	0.0537 (8)	0.0889 (13)	-0.0040 (9)	-0.0184 (12)	0.0056 (8)
C7	0.0688 (15)	0.0610 (12)	0.0650 (14)	-0.0041 (14)	0.0017 (15)	0.0057 (12)

## supplementary materials

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C11	0.0587 (13)	0.0531 (11)	0.0598 (14)	-0.0041 (12)	0.0017 (14)	-0.0040 (9)
C6	0.0613 (14)	0.0544 (11)	0.0548 (14)	-0.0042 (12)	0.0049 (14)	-0.0065 (9)
N2	0.0669 (15)	0.0554 (10)	0.0615 (12)	-0.0027 (10)	-0.0049 (11)	-0.0060 (9)
C1	0.0587 (15)	0.0552 (12)	0.0543 (14)	-0.0048 (10)	-0.0054 (12)	0.0014 (10)
O1	0.0939 (17)	0.0998 (13)	0.0967 (15)	-0.0252 (12)	-0.0188 (13)	-0.0225 (11)
C8	0.087 (2)	0.0523 (12)	0.0808 (19)	-0.0052 (15)	0.0142 (18)	0.0050 (12)
N1	0.0762 (16)	0.0724 (14)	0.0598 (14)	-0.0166 (13)	-0.0058 (12)	-0.0045 (10)
C5	0.0612 (19)	0.0548 (12)	0.0681 (16)	0.0022 (11)	-0.0007 (13)	-0.0027 (11)
C9	0.0749 (18)	0.0668 (15)	0.0813 (19)	-0.0151 (14)	0.0057 (18)	-0.0075 (13)
C4	0.0531 (14)	0.0539 (11)	0.0616 (15)	0.0036 (9)	-0.0017 (12)	-0.0006 (11)
O2	0.1080 (15)	0.0631 (10)	0.0937 (14)	0.0073 (12)	-0.0122 (15)	-0.0038 (9)
C10	0.0635 (15)	0.0672 (14)	0.0706 (16)	-0.0065 (13)	-0.0015 (14)	-0.0088 (13)
C3	0.0599 (17)	0.0611 (13)	0.0816 (19)	0.0080 (13)	-0.0018 (14)	0.0015 (12)
C2	0.0580 (15)	0.0760 (16)	0.0714 (16)	-0.0016 (13)	-0.0133 (13)	0.0070 (13)
C13	0.081 (2)	0.0754 (16)	0.112 (2)	0.0061 (15)	-0.0289 (19)	0.0128 (15)

### *Geometric parameters (Å, °)*

S1—C1	1.698 (2)	C8—H8	0.9300
S1—C4	1.725 (2)	N1—O2	1.228 (3)
O4—C11	1.364 (2)	C5—C4	1.447 (3)
O4—C13	1.408 (3)	C5—H5	0.9300
C7—C8	1.383 (4)	C9—C10	1.383 (3)
C7—C6	1.391 (3)	C9—H9	0.9300
C7—H7	0.9300	C4—C3	1.356 (3)
C11—C10	1.378 (3)	C10—H10	0.9300
C11—C6	1.402 (3)	C3—C2	1.397 (3)
C6—N2	1.414 (3)	C3—H3	0.9300
N2—C5	1.266 (3)	C2—H2	0.9300
C1—C2	1.348 (3)	C13—H13A	0.9600
C1—N1	1.437 (3)	C13—H13B	0.9600
O1—N1	1.224 (3)	C13—H13C	0.9600
C8—C9	1.358 (4)		
C1—S1—C4	89.14 (11)	C4—C5—H5	119.3
C11—O4—C13	117.5 (2)	C8—C9—C10	120.9 (2)
C8—C7—C6	120.3 (2)	C8—C9—H9	119.5
C8—C7—H7	119.9	C10—C9—H9	119.5
C6—C7—H7	119.9	C3—C4—C5	127.9 (2)
O4—C11—C10	124.7 (2)	C3—C4—S1	112.20 (18)
O4—C11—C6	115.5 (2)	C5—C4—S1	119.86 (17)
C10—C11—C6	119.8 (2)	C11—C10—C9	119.9 (2)
C7—C6—C11	119.0 (2)	C11—C10—H10	120.0
C7—C6—N2	123.0 (2)	C9—C10—H10	120.0
C11—C6—N2	117.87 (19)	C4—C3—C2	113.2 (2)
C5—N2—C6	119.9 (2)	C4—C3—H3	123.4
C2—C1—N1	125.7 (2)	C2—C3—H3	123.4
C2—C1—S1	115.04 (17)	C1—C2—C3	110.4 (2)
N1—C1—S1	119.23 (18)	C1—C2—H2	124.8
C9—C8—C7	120.0 (2)	C3—C2—H2	124.8

C9—C8—H8	120.0	O4—C13—H13A	109.5
C7—C8—H8	120.0	O4—C13—H13B	109.5
O1—N1—O2	124.6 (2)	H13A—C13—H13B	109.5
O1—N1—C1	117.6 (2)	O4—C13—H13C	109.5
O2—N1—C1	117.8 (2)	H13A—C13—H13C	109.5
N2—C5—C4	121.3 (2)	H13B—C13—H13C	109.5
N2—C5—H5	119.3		
C13—O4—C11—C10	6.3 (4)	S1—C1—N1—O2	-2.0 (3)
C13—O4—C11—C6	-175.0 (2)	C6—N2—C5—C4	-175.9 (2)
C8—C7—C6—C11	1.5 (4)	C7—C8—C9—C10	0.4 (4)
C8—C7—C6—N2	176.9 (2)	N2—C5—C4—C3	173.5 (3)
O4—C11—C6—C7	-179.7 (2)	N2—C5—C4—S1	-5.6 (3)
C10—C11—C6—C7	-1.0 (3)	C1—S1—C4—C3	0.1 (2)
O4—C11—C6—N2	4.6 (3)	C1—S1—C4—C5	179.4 (2)
C10—C11—C6—N2	-176.7 (2)	O4—C11—C10—C9	178.8 (2)
C7—C6—N2—C5	33.7 (4)	C6—C11—C10—C9	0.2 (4)
C11—C6—N2—C5	-150.8 (2)	C8—C9—C10—C11	0.1 (4)
C4—S1—C1—C2	-0.4 (2)	C5—C4—C3—C2	-179.0 (2)
C4—S1—C1—N1	179.1 (2)	S1—C4—C3—C2	0.2 (3)
C6—C7—C8—C9	-1.2 (4)	N1—C1—C2—C3	-178.9 (2)
C2—C1—N1—O1	-2.5 (4)	S1—C1—C2—C3	0.6 (3)
S1—C1—N1—O1	178.03 (18)	C4—C3—C2—C1	-0.5 (3)
C2—C1—N1—O2	177.5 (2)		

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

$Cg2$  is the centroid of the C6—C11 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10 $\cdots Cg2^i$	0.93	2.77	3.605 (3)	149

Symmetry codes: (i)  $x-1/2, -y+1/2, -z$ .

Fig. 1

