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(Z)-3-Methoxy-N-[(5-nitrothiophen-2-yl)-methylidene]aniline

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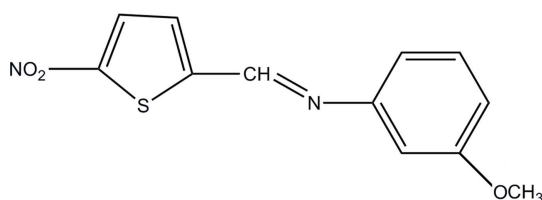
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.030; wR factor = 0.076; data-to-parameter ratio = 14.4.

In the title compound, $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3\text{S}$, the dihedral angle between the benzene and thiophene rings is $43.17(4)^\circ$. The crystal structure is devoid of any hydrogen-bonding interactions. However, π - π interactions between the benzene and thiophene rings [distance between ring centroids = $3.6850(11)$ Å] stack the molecules along the a axis. The absolute structure could not be determined as the crystal studied was a racemic twin with a BASF parameter of 0.31 (6).

Related literature

For biological and industrial properties of Schiff bases, see: Barton & Ollis (1979); Taggi *et al.* (2002). For a related structure, see: Ceylan *et al.* (2011).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3\text{S}$	$V = 1204.51(10)$ Å ³
$M_r = 262.29$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.4612(3)$ Å	$\mu = 0.27$ mm ⁻¹
$b = 10.8737(5)$ Å	$T = 296$ K
$c = 14.8465(9)$ Å	$0.69 \times 0.51 \times 0.28$ mm

Data collection

Stoe IPDS 2 diffractometer	5538 measured reflections
Absorption correction: integration (<i>X-RED</i> ; Stoe & Cie, 2002)	2375 independent reflections
$T_{\min} = 0.873$, $T_{\max} = 0.938$	2200 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	$\Delta\rho_{\max} = 0.17$ e Å ⁻³
$wR(F^2) = 0.076$	$\Delta\rho_{\min} = -0.18$ e Å ⁻³
$S = 1.02$	Absolute structure: Flack (1983),
2375 reflections	986 Friedel pairs
165 parameters	Flack parameter: 0.31 (6)
H-atom parameters constrained	

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) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2561).

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supporting information

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(Z)-3-Methoxy-N-[(5-nitrothiophen-2-yl)methylidene]aniline**Tufan Akbal, Erbil Ađar and Ahmet Erdönmez****S1. Comment**

Schiff bases are used as starting materials in the synthesis of important drugs, such as antibiotics and antiallergic, antiphlogistic, and antitumor substances (Barton & Ollis, 1979). In addition, they have a wide range of industrial applications, such as dyes and pigments (Taggi *et al.*, 2002). Herein we report the synthesis and crystal structure of the title compound.

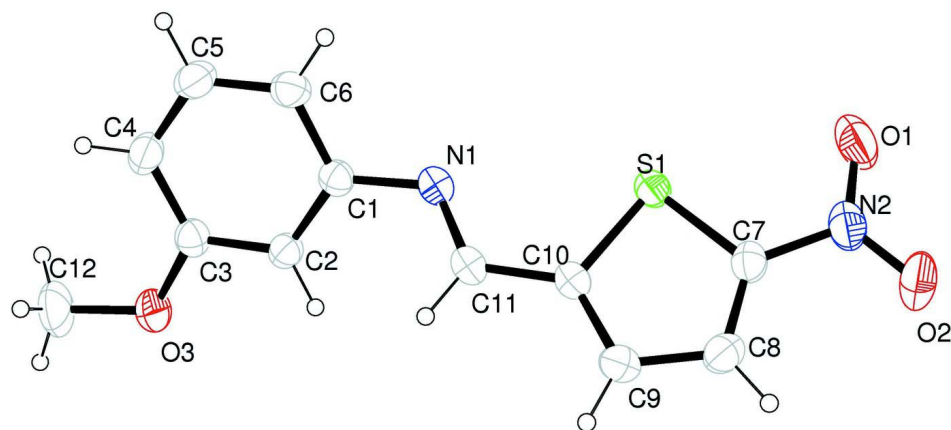
In the title compound (Fig. 1), the dihedral angle between the nitro-thiophene (C7—C10/S1) and the benzene ring (C1—C6) is 47.14 (4) °. The bond distances and angles in the title compound agree very well with the corresponding bond distances and angles reported in a closely related compound (Ceylan *et al.* 2011). The structure is devoid of any hydrogen bonding interactions. However, π — π interactions between the centroids of the benzene and thiophene rings (distance between ring centroids = 3.6850 (11) Å) are observed in the crystal structure.

S2. Experimental

The compound title compound was prepared by refluxing a mixture of a solution of 5-nitro-2-thiophene-carboxaldehyde (0.018 g, 0.120 mmol) in ethanol (20 ml) and a solution of 3-methoxyaniline (0.0142 g, 0.120 mmol) in ethanol (20 ml). The reaction mixture was stirred for 1 h under reflux. The crystals of the title compound suitable for X-ray analysis were obtained from ethanol by slow evaporation (yield %61; m.p 385–386 K).

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 and 0.96 Å, for aryl and methyl H-atoms, respectively. The $U_{\text{iso}}(\text{H})$ were allowed at $1.5U_{\text{eq}}(\text{C methyl})$ or $1.2U_{\text{eq}}(\text{C aryl})$. The compound crystallized as a racemic twin as indicated by *SHELXL97* (Sheldrick, 2008). A twin refinement using the commands TWIN and BASF gave a twin fraction of 0.31 (6)/0.69 (6); 986 Friedel pairs of reflections were not merged.

**Figure 1**

The molecular structure of title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

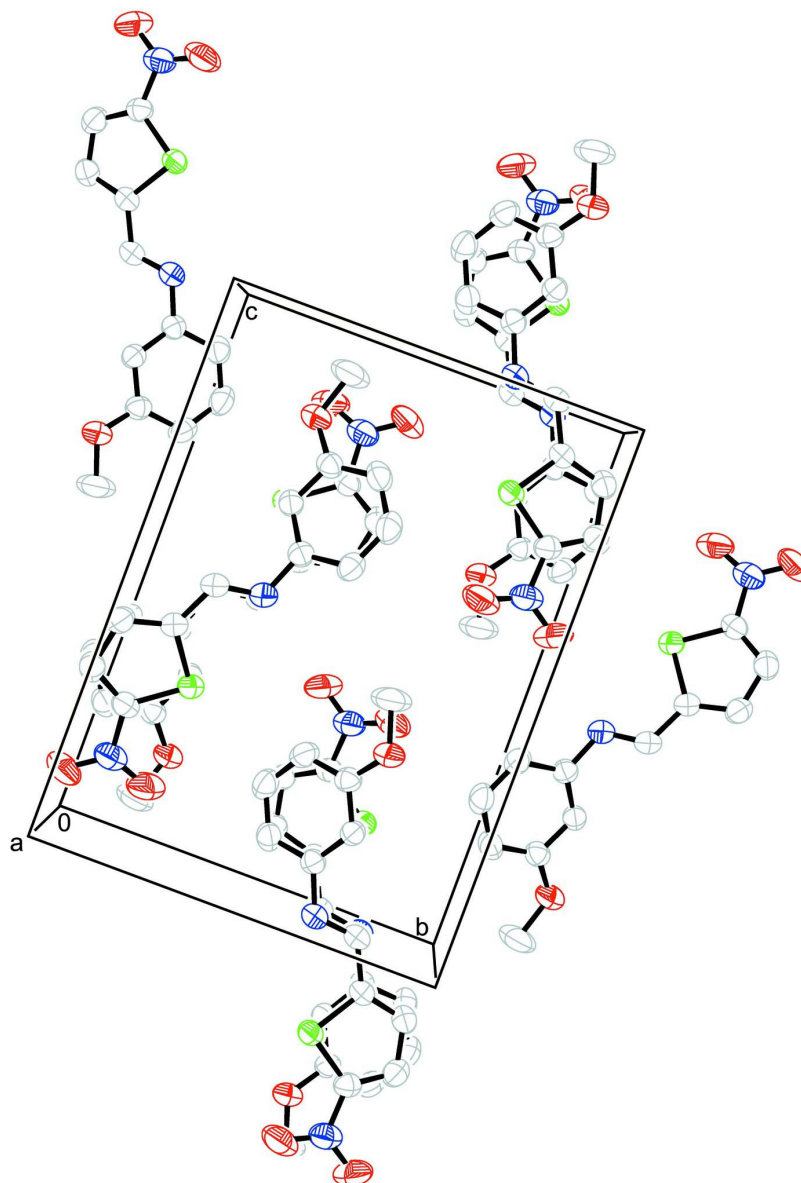


Figure 2

A view of the crystal packing of the title compound. Hydrogen atoms have been excluded for clarity.

(Z)-3-Methoxy-N-[(5-nitrothiophen-2-yl)methylidene]aniline

Crystal data

$C_{12}H_{10}N_2O_3S$

$M_r = 262.29$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.4612 (3) \text{ \AA}$

$b = 10.8737 (5) \text{ \AA}$

$c = 14.8465 (9) \text{ \AA}$

$V = 1204.51 (10) \text{ \AA}^3$

$Z = 4$

$F(000) = 544$

$D_x = 1.446 \text{ Mg m}^{-3}$

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

Cell parameters from 5538 reflections

$\theta = 2.3\text{--}26^\circ$

$\mu = 0.27 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism, yellow

$0.69 \times 0.51 \times 0.28 \text{ mm}$

Data collection

Stoe IPDS 2
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω -scan rotation
 Absorption correction: integration
 (*X-RED*; Stoe & Cie, 2002)
 $T_{\min} = 0.873$, $T_{\max} = 0.938$

5538 measured reflections
 2375 independent reflections
 2200 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -8 \rightarrow 9$
 $k = -13 \rightarrow 13$
 $l = -15 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.076$
 $S = 1.02$
 2375 reflections
 165 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0097 (18)
 Absolute structure: Flack (1983), 986 Friedel
 pairs
 Absolute structure parameter: 0.31 (6)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.17537 (6)	0.72680 (4)	0.17461 (3)	0.04740 (13)
O3	0.2561 (2)	0.78260 (13)	-0.33068 (9)	0.0645 (3)
N1	0.1374 (2)	0.81303 (13)	-0.01636 (9)	0.0475 (3)
N2	0.1792 (2)	0.60779 (17)	0.33474 (11)	0.0665 (4)
O1	0.2270 (3)	0.70702 (19)	0.36455 (10)	0.0961 (6)
C1	0.1284 (2)	0.86180 (14)	-0.10418 (11)	0.0440 (4)
C3	0.1854 (2)	0.85202 (15)	-0.26249 (11)	0.0479 (4)
C7	0.1414 (2)	0.60021 (16)	0.24090 (12)	0.0490 (4)
C10	0.1066 (2)	0.64354 (15)	0.08297 (11)	0.0449 (4)
C4	0.1172 (3)	0.96946 (18)	-0.27339 (13)	0.0585 (5)
H4	0.1131	1.0056	-0.3301	0.070*
C6	0.0617 (3)	0.98144 (16)	-0.11459 (13)	0.0539 (4)
H6	0.0224	1.0258	-0.0648	0.065*

O2	0.1619 (3)	0.51571 (17)	0.38055 (10)	0.0973 (6)
C8	0.0809 (3)	0.50020 (17)	0.19685 (13)	0.0560 (5)
H8	0.0555	0.4250	0.2238	0.067*
C5	0.0552 (3)	1.03209 (18)	-0.19860 (14)	0.0619 (5)
H5	0.0079	1.1106	-0.2057	0.074*
C2	0.1906 (2)	0.79816 (13)	-0.17871 (11)	0.0442 (3)
H2	0.2360	0.7190	-0.1721	0.053*
C11	0.0970 (2)	0.70084 (16)	-0.00462 (11)	0.0471 (4)
H11	0.0602	0.6542	-0.0538	0.057*
C9	0.0616 (3)	0.52531 (17)	0.10520 (13)	0.0546 (4)
H9	0.0222	0.4676	0.0634	0.065*
C12	0.2302 (4)	0.8228 (3)	-0.42063 (14)	0.0905 (8)
H12A	0.2864	0.7659	-0.4612	0.136*
H12B	0.2824	0.9028	-0.4281	0.136*
H12C	0.1042	0.8268	-0.4334	0.136*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0552 (2)	0.0474 (2)	0.03953 (19)	-0.00386 (17)	0.00184 (18)	-0.00330 (17)
O3	0.0855 (9)	0.0682 (8)	0.0397 (6)	0.0033 (7)	0.0063 (6)	0.0038 (6)
N1	0.0494 (7)	0.0529 (7)	0.0401 (7)	-0.0007 (6)	-0.0015 (6)	-0.0038 (6)
N2	0.0752 (11)	0.0795 (10)	0.0447 (8)	0.0042 (9)	0.0000 (9)	0.0045 (8)
O1	0.1360 (17)	0.1054 (13)	0.0468 (8)	-0.0233 (12)	-0.0092 (9)	-0.0107 (9)
C1	0.0418 (8)	0.0459 (8)	0.0444 (9)	-0.0028 (7)	-0.0040 (7)	-0.0006 (7)
C3	0.0481 (9)	0.0512 (8)	0.0443 (8)	-0.0065 (8)	-0.0015 (8)	0.0033 (7)
C7	0.0494 (9)	0.0555 (9)	0.0421 (8)	0.0057 (8)	0.0031 (7)	0.0042 (7)
C10	0.0449 (9)	0.0496 (8)	0.0403 (8)	0.0003 (7)	0.0005 (7)	-0.0045 (7)
C4	0.0659 (11)	0.0538 (9)	0.0558 (10)	-0.0045 (9)	-0.0125 (9)	0.0135 (9)
C6	0.0565 (10)	0.0463 (9)	0.0590 (11)	0.0008 (8)	-0.0086 (9)	-0.0075 (8)
O2	0.1395 (16)	0.0977 (11)	0.0546 (9)	0.0113 (13)	0.0039 (10)	0.0283 (9)
C8	0.0616 (11)	0.0467 (9)	0.0598 (11)	0.0012 (7)	0.0033 (9)	0.0068 (8)
C5	0.0670 (11)	0.0445 (9)	0.0742 (13)	0.0061 (8)	-0.0134 (10)	0.0034 (9)
C2	0.0451 (8)	0.0418 (7)	0.0456 (8)	-0.0014 (6)	-0.0029 (7)	0.0026 (7)
C11	0.0485 (9)	0.0546 (9)	0.0382 (8)	-0.0010 (7)	-0.0002 (7)	-0.0056 (7)
C9	0.0621 (10)	0.0482 (9)	0.0534 (10)	0.0002 (8)	-0.0032 (9)	-0.0078 (8)
C12	0.118 (2)	0.1166 (19)	0.0373 (10)	0.0071 (17)	0.0024 (12)	0.0116 (12)

Geometric parameters (Å, °)

S1—C7	1.7110 (18)	C10—C11	1.444 (2)
S1—C10	1.7128 (17)	C4—C5	1.382 (3)
O3—C3	1.369 (2)	C4—H4	0.9300
O3—C12	1.418 (3)	C6—C5	1.364 (3)
N1—C11	1.269 (2)	C6—H6	0.9300
N1—C1	1.409 (2)	C8—C9	1.395 (3)
N2—O2	1.217 (2)	C8—H8	0.9300
N2—O1	1.220 (2)	C5—H5	0.9300

N2—C7	1.424 (2)	C2—H2	0.9300
C1—C2	1.385 (2)	C11—H11	0.9300
C1—C6	1.401 (2)	C9—H9	0.9300
C3—C2	1.375 (2)	C12—H12A	0.9600
C3—C4	1.384 (3)	C12—H12B	0.9600
C7—C8	1.347 (3)	C12—H12C	0.9600
C10—C9	1.369 (3)		
C7—S1—C10	89.27 (8)	C1—C6—H6	120.4
C3—O3—C12	118.30 (17)	C7—C8—C9	110.49 (17)
C11—N1—C1	118.54 (14)	C7—C8—H8	124.8
O2—N2—O1	123.77 (18)	C9—C8—H8	124.8
O2—N2—C7	118.57 (18)	C6—C5—C4	121.58 (17)
O1—N2—C7	117.66 (17)	C6—C5—H5	119.2
C2—C1—C6	119.65 (16)	C4—C5—H5	119.2
C2—C1—N1	122.36 (14)	C3—C2—C1	120.01 (14)
C6—C1—N1	117.92 (15)	C3—C2—H2	120.0
O3—C3—C2	115.04 (15)	C1—C2—H2	120.0
O3—C3—C4	124.34 (16)	N1—C11—C10	121.81 (15)
C2—C3—C4	120.60 (16)	N1—C11—H11	119.1
C8—C7—N2	126.02 (17)	C10—C11—H11	119.1
C8—C7—S1	114.83 (13)	C10—C9—C8	113.18 (17)
N2—C7—S1	119.15 (14)	C10—C9—H9	123.4
C9—C10—C11	127.61 (16)	C8—C9—H9	123.4
C9—C10—S1	112.23 (14)	O3—C12—H12A	109.5
C11—C10—S1	120.14 (12)	O3—C12—H12B	109.5
C5—C4—C3	118.92 (17)	H12A—C12—H12B	109.5
C5—C4—H4	120.5	O3—C12—H12C	109.5
C3—C4—H4	120.5	H12A—C12—H12C	109.5
C5—C6—C1	119.22 (18)	H12B—C12—H12C	109.5
C5—C6—H6	120.4		
C11—N1—C1—C2	-42.6 (2)	N1—C1—C6—C5	178.55 (16)
C11—N1—C1—C6	140.62 (16)	N2—C7—C8—C9	-179.03 (19)
C12—O3—C3—C2	170.13 (19)	S1—C7—C8—C9	0.5 (2)
C12—O3—C3—C4	-11.7 (3)	C1—C6—C5—C4	-1.7 (3)
O2—N2—C7—C8	2.6 (3)	C3—C4—C5—C6	0.8 (3)
O1—N2—C7—C8	-177.3 (2)	O3—C3—C2—C1	177.79 (15)
O2—N2—C7—S1	-176.88 (17)	C4—C3—C2—C1	-0.4 (3)
O1—N2—C7—S1	3.2 (3)	C6—C1—C2—C3	-0.6 (2)
C10—S1—C7—C8	-0.21 (15)	N1—C1—C2—C3	-177.34 (15)
C10—S1—C7—N2	179.32 (16)	C1—N1—C11—C10	178.65 (15)
C7—S1—C10—C9	-0.11 (15)	C9—C10—C11—N1	177.72 (17)
C7—S1—C10—C11	178.33 (15)	S1—C10—C11—N1	-0.5 (2)
O3—C3—C4—C5	-177.69 (18)	C11—C10—C9—C8	-177.90 (17)
C2—C3—C4—C5	0.3 (3)	S1—C10—C9—C8	0.4 (2)
C2—C1—C6—C5	1.6 (3)	C7—C8—C9—C10	-0.5 (2)