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4-Methoxy-*N*-[(*E*)-(5-nitrothiophen-2-yl)-methylidene]aniline

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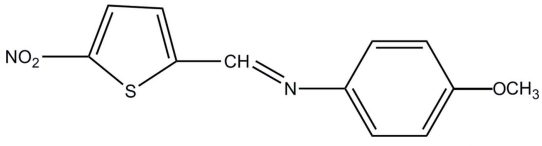
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.125; data-to-parameter ratio = 17.3.

The title molecule, $C_{12}H_{10}N_2O_3S$, is nonplanar with an interplanar angle of 33.44 (7)° between the benzene and thiophene rings. In the crystal there exist only weak intermolecular C—H···O interactions, π–π interactions between the benzene rings [centroid–centroid distance = 3.7465 (14) Å] and X—Y···π interactions to the thiophene and benzene rings [N···centroid distances = 3.718 (3) and 3.355 (3) Å, respectively]. Intermolecular C—H···O interactions link the molecules into chains parallel to the a axis.

Related literature

For the biological properties of Schiff bases, see Layer (1963); Ingold (1969); Barton & Ollis (1979). For the application of Schiff bases in industry, see Taggi *et al.* (2002). For related structures, see Ceylan *et al.* (2011); Özdemir & Işık (2012).



Experimental

Crystal data

$C_{12}H_{10}N_2O_3S$
M_r = 262.28
Monoclinic, $P2_1/c$
a = 12.5641 (7) Å
b = 13.1441 (5) Å
c = 7.7896 (4) Å
β = 106.012 (4)°
V = 1236.50 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 296 K
0.69 × 0.51 × 0.28 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.873, T_{max} = 0.938
20097 measured reflections
2841 independent reflections
2076 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.15
2841 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.93	2.48	3.292 (3)	146
C9—H9···O3^{ii}	0.93	2.40	3.273 (3)	156

Symmetry codes: (i) x, y, $z+1$; (ii) $x+1$, y, 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: *WinGX* (Farrugia, 1997) and *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: FB2260).

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supporting information

Acta Cryst. (2012). E**68**, o3026 [https://doi.org/10.1107/S1600536812034344]

4-Methoxy-*N*-[(*E*)-(5-nitrothiophen-2-yl)methylidene]aniline

Tufan Akbal, Erbil Ağar, Sümeyye Gümüş and Ahmet Erdönmez

S1. Comment

The Schiff bases, *i. e.* the 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 (Layer, 1963; Ingold, 1969; Barton & Ollis, 1979). On the industrial scale, they have a wide range of applications, such as dyes and pigments (Taggi *et al.*, 2002).

The title molecule is shown in Fig. 1. The molecule is non-planar, with an interplanar angle of 33.44 (7)° between the benzene and the substituted thiophene rings. The length of the C11═N2 double bond is 1.268 (3) Å. This value agrees well with the analogous bond reported elsewhere (Ceylan *et al.* 2011; Özdemir Tari & Işık, 2012). In the crystal (Fig. 2), two different C—H···O intermolecular interactions (Table 1) generate chains of molecules extending along the *a* axis. The distance 3.7465 (14) Å between the centroids of the neighbouring benzene rings related by the symmetry operation 1-*x*, -*y*, 1-*z* indicates a *π*-electron—*π*-electron ring interaction. Intermolecular X—Y···*π*-electron ring interactions are also present in the crystal structure (N1—O1···*Cg*1^{i}=3.718 (3) Å and N1—O2···*Cg*2^{ii}=3.355 (3) Å where *Cg*1 and *Cg*2 are the centroids of the rings C7—C10/S1 (substituted thiophene) and C1—C6 (benzene), respectively, and the symmetry codes i and ii correspond to 2-*x*, -*y*, -*z* and *x*, 1/2-*y*, -1/2+*z*, respectively.

S2. Experimental

The title compound was prepared under reflux at room temperature of a mixture of two solutions: One contained 5-nitro-2-thiophene-carboxaldehyde (0.016 g, 0.100 mmol) in 20 ml of absolute ethanol, while the other 4-methoxyaniline (0.012 g, 0.100 mmol) in 20 ml of absolute ethanol. The reaction mixture was stirred for 1 h under reflux. Yellow, transparent crystals were obtained by slow evaporation from ethanol solution at room temperature (yield 68 wt. %; m.p. 414–417 K). The average size of the prismatic crystals was about 0.12 × 0.45 × 0.80 mm.

S3. Refinement

All the hydrogen atoms appeared in the difference electron density map, nevertheless, they were situated into the idealized positions and refined in the riding atom formalism. The applied constraints: C_{methyl}—H_{methyl}=0.96, C_{aryl}—H_{aryl}=0.93 Å. $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$, $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$.

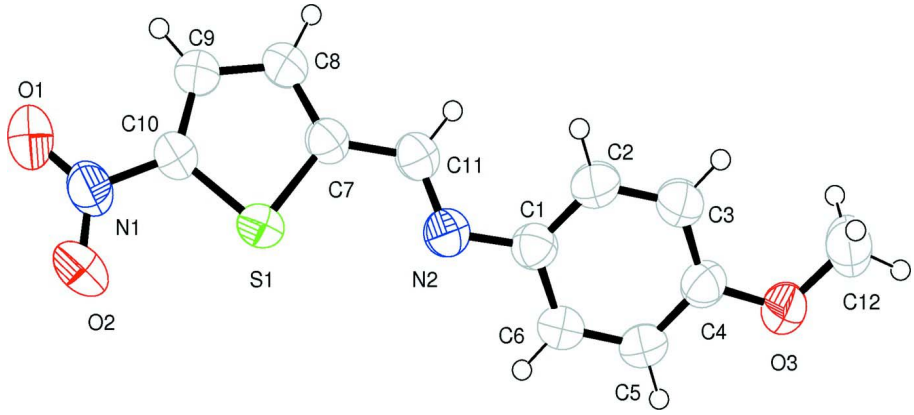

Figure 1

The title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 30% probability level.

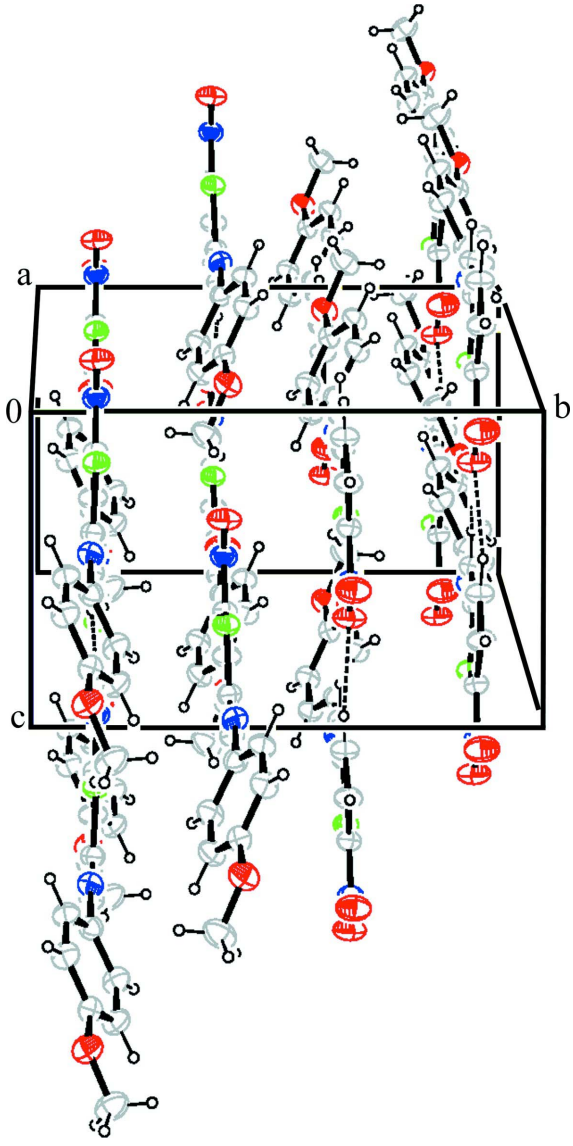

Figure 2

A view of the crystal packing of the title compound.

4-Methoxy-*N*-[(*E*)-(5-nitrothiophen-2-yl)methylidene]aniline

Crystal data

$C_{12}H_{10}N_2O_3S$
$M_r = 262.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.5641\ (7)$ Å
$b = 13.1441\ (5)$ Å
$c = 7.7896\ (4)$ Å
$\beta = 106.012\ (4)°$
$V = 1236.50\ (10)$ Å^3
$Z = 4$

$F(000) = 544$
$D_x = 1.409$ Mg m^{-3}
Melting point = 414–417 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20097 reflections
$\theta = 2.3–27.6°$
$\mu = 0.26$ mm^{-1}
$T = 296$ K
Prism, yellow
0.69 × 0.51 × 0.28 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
w–scan rotation
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.873, T_{max} = 0.938
20097 measured reflections
2841 independent reflections
2076 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.6°, θ_{min} = 2.3°
h = −16→16
k = −17→17
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.15
2841 reflections
164 parameters
0 restraints
39 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.3031P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.77562 (5)	0.12622 (5)	0.06150 (7)	0.05989 (19)
C1	0.51607 (18)	0.11596 (17)	0.3360 (3)	0.0561 (5)
N2	0.60693 (16)	0.11489 (15)	0.2619 (3)	0.0597 (5)
C11	0.70493 (19)	0.11725 (18)	0.3637 (3)	0.0586 (5)
H11	0.7168	0.1176	0.4870	0.070*
C9	0.97699 (19)	0.1197 (2)	0.2631 (3)	0.0658 (6)
H9	1.0540	0.1186	0.3003	0.079*
O3	0.23334 (13)	0.11016 (14)	0.5127 (2)	0.0722 (5)
N1	0.95778 (19)	0.12583 (16)	−0.0612 (3)	0.0693 (5)
C10	0.91555 (18)	0.12453 (18)	0.0913 (3)	0.0576 (5)
C4	0.32882 (18)	0.11543 (17)	0.4617 (3)	0.0572 (5)
O1	1.05738 (18)	0.12534 (19)	−0.0377 (3)	0.0968 (7)
O2	0.88984 (18)	0.12695 (18)	−0.2095 (2)	0.0922 (6)
C7	0.79851 (18)	0.11941 (18)	0.2889 (3)	0.0554 (5)
C8	0.90855 (19)	0.1166 (2)	0.3776 (3)	0.0657 (6)

H8	0.9353	0.1131	0.5013	0.079*
C2	0.5164 (2)	0.1630 (2)	0.4951 (3)	0.0660 (6)
H2	0.5804	0.1956	0.5607	0.079*
C3	0.4248 (2)	0.1628 (2)	0.5585 (3)	0.0661 (6)
H3	0.4273	0.1944	0.6663	0.079*
C5	0.32604 (19)	0.0703 (2)	0.3006 (3)	0.0643 (6)
H5	0.2614	0.0390	0.2339	0.077*
C6	0.41788 (18)	0.0713 (2)	0.2382 (3)	0.0616 (6)
H6	0.4144	0.0416	0.1285	0.074*
C12	0.2326 (3)	0.1537 (3)	0.6792 (4)	0.0895 (9)
H12A	0.1627	0.1402	0.7023	0.134*
H12B	0.2435	0.2259	0.6754	0.134*
H12C	0.2911	0.1245	0.7725	0.134*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0572 (3)	0.0756 (4)	0.0446 (3)	−0.0047 (3)	0.0102 (2)	−0.0023 (3)
C1	0.0560 (12)	0.0581 (13)	0.0533 (12)	−0.0017 (10)	0.0132 (10)	0.0017 (10)
N2	0.0581 (11)	0.0657 (12)	0.0558 (11)	−0.0027 (9)	0.0163 (9)	0.0021 (9)
C11	0.0615 (13)	0.0663 (14)	0.0499 (12)	−0.0029 (11)	0.0184 (10)	0.0034 (11)
C9	0.0530 (12)	0.0927 (18)	0.0504 (13)	−0.0001 (12)	0.0120 (10)	−0.0022 (12)
O3	0.0560 (9)	0.0878 (12)	0.0758 (11)	−0.0055 (8)	0.0235 (8)	−0.0134 (9)
N1	0.0777 (14)	0.0796 (14)	0.0574 (13)	−0.0052 (12)	0.0302 (11)	−0.0056 (11)
C10	0.0595 (12)	0.0671 (13)	0.0487 (12)	−0.0024 (11)	0.0193 (10)	−0.0034 (11)
C4	0.0505 (11)	0.0578 (13)	0.0617 (14)	0.0034 (10)	0.0129 (10)	−0.0004 (11)
O1	0.0818 (13)	0.1392 (19)	0.0829 (14)	−0.0052 (13)	0.0452 (11)	−0.0052 (13)
O2	0.1034 (14)	0.1289 (18)	0.0462 (10)	−0.0080 (13)	0.0236 (10)	−0.0058 (11)
C7	0.0579 (12)	0.0619 (13)	0.0471 (12)	−0.0052 (10)	0.0158 (9)	0.0001 (10)
C8	0.0590 (13)	0.0936 (18)	0.0433 (12)	−0.0005 (12)	0.0122 (10)	−0.0002 (12)
C2	0.0553 (13)	0.0729 (15)	0.0680 (15)	−0.0135 (11)	0.0141 (11)	−0.0142 (12)
C3	0.0628 (14)	0.0727 (15)	0.0639 (15)	−0.0098 (12)	0.0192 (12)	−0.0175 (12)
C5	0.0508 (12)	0.0765 (16)	0.0612 (15)	−0.0081 (11)	0.0082 (10)	−0.0097 (12)
C6	0.0572 (13)	0.0746 (16)	0.0500 (13)	−0.0030 (11)	0.0099 (10)	−0.0063 (11)
C12	0.0811 (18)	0.106 (2)	0.094 (2)	−0.0108 (16)	0.0447 (16)	−0.0271 (17)

Geometric parameters (Å, º)

S1—C10	1.709 (2)	N1—C10	1.429 (3)
S1—C7	1.717 (2)	C4—C5	1.380 (3)
C1—C2	1.384 (3)	C4—C3	1.381 (3)
C1—C6	1.389 (3)	C7—C8	1.365 (3)
C1—N2	1.414 (3)	C8—H8	0.9300
N2—C11	1.268 (3)	C2—C3	1.372 (3)
C11—C7	1.449 (3)	C2—H2	0.9300
C11—H11	0.9300	C3—H3	0.9300
C9—C10	1.350 (3)	C5—C6	1.370 (3)
C9—C8	1.400 (3)	C5—H5	0.9300

C9—H9	0.9300	C6—H6	0.9300
O3—C4	1.366 (3)	C12—H12A	0.9600
O3—C12	1.420 (3)	C12—H12B	0.9600
N1—O1	1.214 (3)	C12—H12C	0.9600
N1—O2	1.232 (3)		

C10—S1—C7	89.19 (10)	C11—C7—S1	119.49 (17)
C2—C1—C6	117.6 (2)	C7—C8—C9	113.0 (2)
C2—C1—N2	124.5 (2)	C7—C8—H8	123.5
C6—C1—N2	117.8 (2)	C9—C8—H8	123.5
C11—N2—C1	119.9 (2)	C3—C2—C1	121.7 (2)
N2—C11—C7	120.3 (2)	C3—C2—H2	119.2
N2—C11—H11	119.9	C1—C2—H2	119.2
C7—C11—H11	119.9	C2—C3—C4	119.8 (2)
C10—C9—C8	110.4 (2)	C2—C3—H3	120.1
C10—C9—H9	124.8	C4—C3—H3	120.1
C8—C9—H9	124.8	C6—C5—C4	120.4 (2)
C4—O3—C12	118.2 (2)	C6—C5—H5	119.8
O1—N1—O2	124.1 (2)	C4—C5—H5	119.8
O1—N1—C10	118.6 (2)	C5—C6—C1	121.2 (2)
O2—N1—C10	117.3 (2)	C5—C6—H6	119.4
C9—C10—N1	125.7 (2)	C1—C6—H6	119.4
C9—C10—S1	114.89 (17)	O3—C12—H12A	109.5
N1—C10—S1	119.41 (18)	O3—C12—H12B	109.5
O3—C4—C5	116.0 (2)	H12A—C12—H12B	109.5
O3—C4—C3	124.7 (2)	O3—C12—H12C	109.5
C5—C4—C3	119.3 (2)	H12A—C12—H12C	109.5
C8—C7—C11	128.1 (2)	H12B—C12—H12C	109.5
C8—C7—S1	112.44 (16)		

C2—C1—N2—C11	30.4 (4)	C10—S1—C7—C8	0.3 (2)
C6—C1—N2—C11	−153.0 (2)	C10—S1—C7—C11	−179.7 (2)
C1—N2—C11—C7	−178.1 (2)	C11—C7—C8—C9	179.8 (2)
C8—C9—C10—N1	−178.2 (2)	S1—C7—C8—C9	−0.2 (3)
C8—C9—C10—S1	0.3 (3)	C10—C9—C8—C7	−0.1 (3)
O1—N1—C10—C9	−2.1 (4)	C6—C1—C2—C3	2.4 (4)
O2—N1—C10—C9	177.6 (3)	N2—C1—C2—C3	178.9 (2)
O1—N1—C10—S1	179.5 (2)	C1—C2—C3—C4	−0.6 (4)
O2—N1—C10—S1	−0.8 (3)	O3—C4—C3—C2	179.6 (2)
C7—S1—C10—C9	−0.4 (2)	C5—C4—C3—C2	−1.0 (4)
C7—S1—C10—N1	178.2 (2)	O3—C4—C5—C6	−179.8 (2)
C12—O3—C4—C5	178.8 (2)	C3—C4—C5—C6	0.8 (4)
C12—O3—C4—C3	−1.8 (4)	C4—C5—C6—C1	1.0 (4)
N2—C11—C7—C8	−176.2 (2)	C2—C1—C6—C5	−2.6 (4)
N2—C11—C7—S1	3.8 (3)	N2—C1—C6—C5	−179.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.93	2.48	3.292 (3)	146
C9—H9···O3^{ii}	0.93	2.40	3.273 (3)	156

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*+1, *y*, *z*.