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(*E*)-1-(5-Methylthiophen-2-yl)-*N*-(4-nitrophenyl)-methanimine

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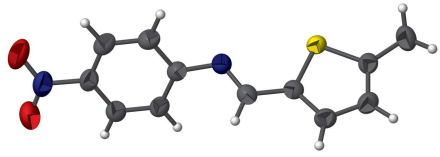
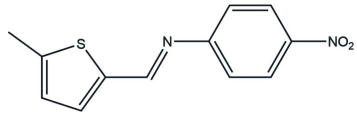
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{12}H_{10}N_2O_2S$, was synthesized *via* the acid-catalyzed condensation of 4-nitroaniline and 5-methyl-2-thiophenecarboxaldehyde in a methanol–water solution. The dihedral angle between the benzene and thiophene rings is 54.62 (3)°. No directional interactions could be identified in the extended structure.

3D view



Chemical scheme



Structure description

The molecular structure of the title compound is shown in Fig. 1. The methylthiophene group makes a dihedral angle of 54.62 (3)° with respect to the plane defined by the nitroaniline ring. Within the aniline ring, the nitro group is nearly coplanar with the phenyl ring [C9—C10—N2—O2 = 3.47 (4)°]. This slight twist of the nitro group is not as large as the 9.0 (3)° angle in (*E*)-*N*-(4-nitrophenyl)-1-(thiophen-2-yl)methanimine (Asiri *et al.*, 2012). In the extended structure, no significant intermolecular interactions occur. A view of the unit cell along [010] is shown in Fig. 2.

Synthesis and crystallization

In a 50-ml Erlenmeyer flask, 1.26 g of 5-methyl-2-thiophene carboxaldehyde (10.0 mmol) were added to a solution of 10 ml of H_2O and 20 ml of a 2.5 mmol l^{-1} solution of NH_4HF_2 in methanol (Lassagne *et al.*, 2015). After swirling the solution to mix the liquids, 1.38 g (10.0 mmol) of 4-nitroaniline were added. The solution was stirred for 24 h even though a yellow precipitate had formed after 4 h. The resulting solid was filtered and washed with cold water then dried (2.03 g; 82%). Crystals were grown from CH_2Cl_2 solutions. Data: m.p. 429 K; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 2.59 (*s*, 3H), 6.86 (*dd*, 1H), 7.26 (*m*, 2H), 7.39 (*d*, 1H), 8.26 (*m*, 2H), 8.46 (*s*, 1H); ^{13}C NMR ($CDCl_3$, 300 MHz): δ = 16.1, 121.5, 125.0,

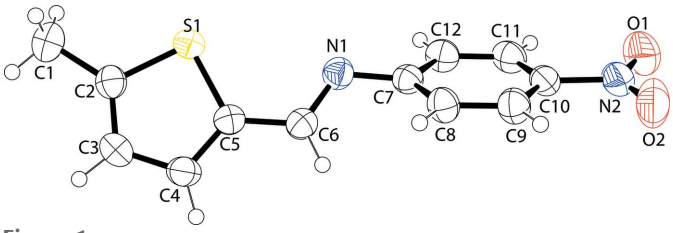


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

126.7, 134.6, 139.8, 145.4, 148.1, 155.1, 157.4; ATR FTIR (cm^{-1}): 3398 (*w*), 2438 (*s*), 1610 (*s*), 1598 (*s*), 1503 (*s*), 1406 (*s*), 1198 (*s*), 1163 (*s*), 1106 (*s*), 1054 (*s*), 967 (*s*), 870 (*s*), 856 (*s*).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

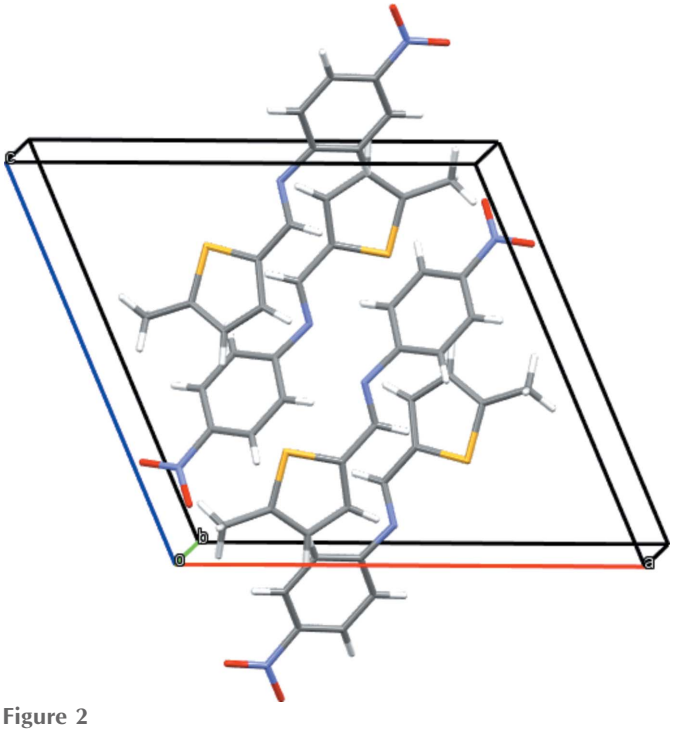


Figure 2
A view of the unit-cell packing along [010].

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}N_2O_2S$
M_r	246.28
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	13.5979 (7), 7.3148 (3), 12.5931 (6)
β (°)	112.763 (5)
V (Å^3)	1155.04 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.27
Crystal size (mm)	0.34 × 0.32 × 0.19
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.796, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28914, 4318, 3467
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.781
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.123, 1.07
No. of reflections	4318
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.33

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *OLEX2* (Bourhis *et al.*, 2015).

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x191601 [https://doi.org/10.1107/S2414314619016018]

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Crystal data

$C_{12}H_{10}N_2O_2S$
M_r = 246.28
Monoclinic, $P2_1/c$
a = 13.5979 (7) Å
b = 7.3148 (3) Å
c = 12.5931 (6) Å
β = 112.763 (5)°
V = 1155.04 (9) Å^3
Z = 4
$F(000)$ = 512
D_x = 1.416 Mg m^{-3}
Melting point: 429 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8567 reflections
θ = 4.5–32.7°
μ = 0.27 mm^{-1}
T = 293 K
Plate, yellow
0.34 × 0.32 × 0.19 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1790 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 2009)
T_{min} = 0.796, T_{max} = 1.000
28914 measured reflections
4318 independent reflections
3467 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 33.7°, θ_{min} = 4.3°
h = −20→21
k = −11→11
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.07
4318 reflections
155 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.2252P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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. H atoms were included in calculated positions with C—H distances of 0.93 Å and 0.96 Å based upon type of carbon atom and were included in the refinement in riding motion approximation with $U_{iso} = 1.2U_{eq}$(carrier) or $1.5U_{eq}$(methyl carrier).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.86901 (12)	0.5813 (3)	0.40834 (15)	0.0629 (4)
H1A	0.9087	0.6247	0.4850	0.094*
H1B	0.9003	0.6280	0.3574	0.094*
H1C	0.8706	0.4501	0.4077	0.094*
C2	0.75619 (10)	0.64523 (16)	0.36931 (11)	0.0420 (2)
C3	0.70532 (11)	0.71403 (19)	0.43482 (11)	0.0472 (3)
H3	0.7381	0.7283	0.5142	0.057*
C4	0.59850 (11)	0.76142 (18)	0.37063 (11)	0.0454 (3)
H4	0.5532	0.8095	0.4031	0.054*
C5	0.56820 (9)	0.72928 (16)	0.25515 (10)	0.0383 (2)
C6	0.46547 (10)	0.76359 (16)	0.16459 (11)	0.0399 (2)
H6	0.4091	0.8003	0.1840	0.048*
C7	0.34681 (10)	0.76957 (16)	−0.02564 (10)	0.0387 (2)
C8	0.25773 (11)	0.68505 (19)	−0.01918 (11)	0.0450 (3)
H8	0.2649	0.6153	0.0452	0.054*
C9	0.15877 (11)	0.7036 (2)	−0.10729 (12)	0.0485 (3)
H9	0.0991	0.6471	−0.1031	0.058*
C10	0.15028 (11)	0.80772 (18)	−0.20170 (11)	0.0449 (3)
C11	0.23669 (12)	0.89611 (19)	−0.20981 (11)	0.0485 (3)
H11	0.2285	0.9684	−0.2734	0.058*
C12	0.33524 (11)	0.87551 (18)	−0.12212 (12)	0.0465 (3)
H12	0.3945	0.9324	−0.1271	0.056*
N1	0.44994 (9)	0.74494 (15)	0.05856 (9)	0.0433 (2)
N2	0.04664 (11)	0.8230 (2)	−0.29732 (11)	0.0588 (3)
O1	0.04178 (11)	0.9053 (2)	−0.38389 (10)	0.0808 (4)
O2	−0.03044 (10)	0.7504 (3)	−0.28855 (12)	0.0869 (4)
S1	0.67202 (2)	0.63767 (4)	0.22616 (3)	0.04172 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (7)	0.0756 (10)	0.0608 (9)	0.0099 (7)	0.0119 (6)	0.0015 (8)
C2	0.0425 (6)	0.0397 (6)	0.0415 (6)	−0.0008 (4)	0.0138 (5)	0.0025 (4)
C3	0.0525 (7)	0.0503 (7)	0.0366 (6)	−0.0037 (5)	0.0149 (5)	−0.0031 (5)
C4	0.0483 (6)	0.0487 (6)	0.0429 (6)	−0.0010 (5)	0.0217 (5)	−0.0063 (5)
C5	0.0395 (5)	0.0359 (5)	0.0411 (5)	−0.0014 (4)	0.0173 (4)	−0.0012 (4)
C6	0.0392 (5)	0.0362 (5)	0.0448 (6)	−0.0008 (4)	0.0167 (5)	−0.0014 (4)
C7	0.0429 (6)	0.0353 (5)	0.0384 (5)	0.0025 (4)	0.0163 (4)	0.0003 (4)

C8	0.0466 (6)	0.0471 (6)	0.0396 (6)	−0.0020 (5)	0.0150 (5)	0.0058 (5)
C9	0.0444 (6)	0.0545 (7)	0.0451 (6)	−0.0036 (5)	0.0154 (5)	0.0009 (6)
C10	0.0472 (6)	0.0467 (6)	0.0360 (5)	0.0078 (5)	0.0108 (5)	−0.0034 (5)
C11	0.0618 (8)	0.0446 (6)	0.0391 (6)	0.0066 (6)	0.0196 (6)	0.0059 (5)
C12	0.0517 (7)	0.0456 (6)	0.0450 (6)	−0.0013 (5)	0.0218 (5)	0.0047 (5)
N1	0.0415 (5)	0.0451 (5)	0.0427 (5)	0.0026 (4)	0.0157 (4)	0.0029 (4)
N2	0.0564 (7)	0.0685 (8)	0.0422 (6)	0.0143 (6)	0.0088 (5)	−0.0060 (6)
O1	0.0834 (9)	0.0975 (10)	0.0456 (6)	0.0223 (7)	0.0075 (6)	0.0146 (6)
O2	0.0499 (7)	0.1314 (13)	0.0642 (8)	0.0002 (8)	0.0054 (6)	0.0030 (8)
S1	0.04364 (17)	0.04606 (17)	0.03761 (15)	0.00209 (12)	0.01807 (12)	−0.00092 (11)

Geometric parameters (Å, º)

C1—H1A	0.9600	C7—C8	1.3901 (18)
C1—H1B	0.9600	C7—C12	1.3977 (17)
C1—H1C	0.9600	C7—N1	1.4045 (16)
C1—C2	1.4935 (19)	C8—H8	0.9300
C2—C3	1.3616 (19)	C8—C9	1.3801 (18)
C2—S1	1.7222 (13)	C9—H9	0.9300
C3—H3	0.9300	C9—C10	1.3788 (19)
C3—C4	1.405 (2)	C10—C11	1.379 (2)
C4—H4	0.9300	C10—N2	1.4612 (18)
C4—C5	1.3698 (17)	C11—H11	0.9300
C5—C6	1.4429 (17)	C11—C12	1.376 (2)
C5—S1	1.7246 (12)	C12—H12	0.9300
C6—H6	0.9300	N2—O1	1.2247 (19)
C6—N1	1.2760 (17)	N2—O2	1.218 (2)
H1A—C1—H1B	109.5	C8—C7—N1	122.47 (11)
H1A—C1—H1C	109.5	C12—C7—N1	118.10 (11)
H1B—C1—H1C	109.5	C7—C8—H8	119.7
C2—C1—H1A	109.5	C9—C8—C7	120.66 (12)
C2—C1—H1B	109.5	C9—C8—H8	119.7
C2—C1—H1C	109.5	C8—C9—H9	120.7
C1—C2—S1	121.10 (11)	C10—C9—C8	118.54 (12)
C3—C2—C1	127.90 (13)	C10—C9—H9	120.7
C3—C2—S1	111.01 (10)	C9—C10—C11	122.24 (12)
C2—C3—H3	123.3	C9—C10—N2	119.02 (13)
C2—C3—C4	113.39 (12)	C11—C10—N2	118.73 (13)
C4—C3—H3	123.3	C10—C11—H11	120.6
C3—C4—H4	123.6	C12—C11—C10	118.84 (12)
C5—C4—C3	112.81 (12)	C12—C11—H11	120.6
C5—C4—H4	123.6	C7—C12—H12	119.8
C4—C5—C6	127.64 (12)	C11—C12—C7	120.37 (13)
C4—C5—S1	110.91 (10)	C11—C12—H12	119.8
C6—C5—S1	121.45 (9)	C6—N1—C7	119.28 (11)
C5—C6—H6	119.1	O1—N2—C10	118.28 (15)
N1—C6—C5	121.75 (11)	O2—N2—C10	118.78 (14)

N1—C6—H6	119.1	O2—N2—O1	122.91 (14)
C8—C7—C12	119.33 (11)	C2—S1—C5	91.88 (6)