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

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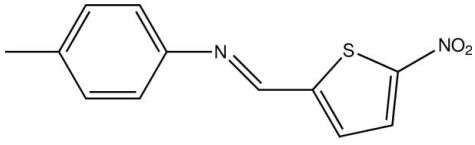
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.063; wR factor = 0.177; data-to-parameter ratio = 14.3.

In the crystal structure of the title compound, $C_{12}H_{10}N_2O_2S$, the benzene and the 2-nitrothiophene rings make a dihedral angle of 7.47 (12)°. The dihedral angle between the nitro group and the attached ring is 1.9 (6)°.

Related literature

For related structures, see: Demirtaş *et al.* (2009); Ceylan *et al.* (2011).



Experimental

Crystal data

$C_{12}H_{10}N_2O_2S$
M_r = 246.28
Monoclinic, $P2_1/n$
a = 4.7661 (4) Å
b = 22.8201 (18) Å
c = 10.7793 (7) Å
β = 92.704 (7)°
V = 1171.08 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 296 K
0.17 × 0.15 × 0.12 mm

Data collection

Oxford Diffraction SuperNova Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.771, T_{max} = 1.000
3997 measured reflections
2211 independent reflections
1549 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.177
S = 1.05
2211 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *WinGX* (Farrugia, 1997) and *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.* 2009) and *ORTEP-3 for Windows* (Farrugia, 1997);; software used to prepare material for publication: OLEX2 (Dolomanov *et al.* 2009), *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: NC2283).

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supplementary materials

Acta Cryst. (2012). E**68**, o2116 [doi:10.1107/S1600536812026062]

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

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Comment

The title compound was by the reaction between 5-nitrothiophene-2-carboxaldehyde and *p*-Toluidine. For the identification of this compound a single crystal structure analysis was performed. It is noted that some 2-nitrothiophene structures have already been reported in literature (Demirtaş *et al.* 2009; Ceylan *et al.* 2011). In the crystal structure the dihedral angle between the 6-membered and the 2-nitrothiophene rings amount to 7.47 (12)° and the torsion angle along C5—N1—C8—C9 is 179.7 (3)°. Both rings are in a trans arrangement with respect to the C-N double bond and the nitro group is oriented almost coplanar to the 2-nitrothiophene plane (Fig. 1).

Experimental

A mixture of 5-nitrothiophene-2-carboxaldehyde (0.011 g 0.066 mmol) in 20 ml e thanol and of *p*-Toluidine (0.007 g 0.066 mmol) in 20 ml ethanol was refluxed for 1 h. Single crystals suitable for X-ray analysis were obtained from a solution of the title compound in ethanol by slow evaporation of the solvent (yield % 78; m.p 101–103 °C).

Refinement

All hydrogen atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip and were refined with U_{iso}(H)= 1.2U_{eq}(C) (1.5 for methyl H atoms using a riding model with C—H = 0.930 for aromatic and and 0.960 for methyl H atoms.

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *WinGX* (Farrugia, 1997) and *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.* 2009) and *ORTEP-3 for Windows* (Farrugia, 1997);; software used to prepare material for publication: OLEX2 (Dolomanov *et al.* 2009), *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

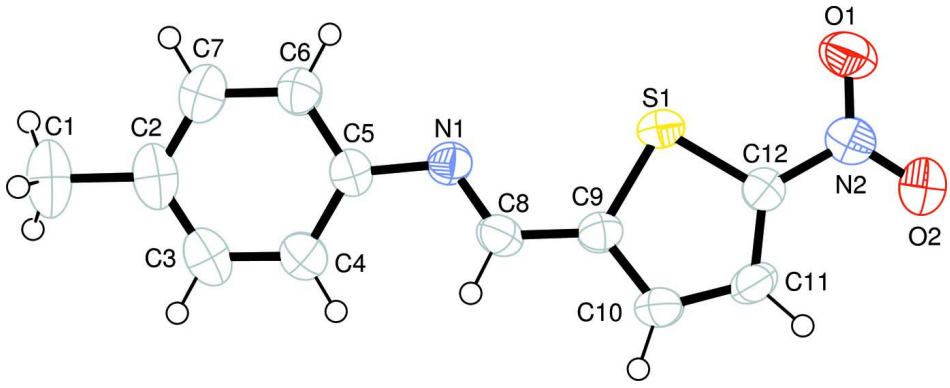

Figure 1

The asymmetric unit of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

$C_{12}H_{10}N_2O_2S$
$M_r = 246.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.7661\ (4)$ Å
$b = 22.8201\ (18)$ Å
$c = 10.7793\ (7)$ Å
$\beta = 92.704\ (7)°$
$V = 1171.08\ (15)$ Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.397$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1442 reflections
$\theta = 3.3–27.6°$
$\mu = 0.26$ mm^{-1}
$T = 296$ K
Prism, brown
0.17 × 0.15 × 0.12 mm

Data collection

Oxford Diffraction SuperNova Eos diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 16.0454 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.771$, $T_{max} = 1.000$
3997 measured reflections
2211 independent reflections
1549 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.3°$
$h = -5\rightarrow5$
$k = -27\rightarrow26$
$l = -13\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.177$
$S = 1.05$
2211 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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}
C1	1.8771 (9)	0.51221 (18)	0.2226 (4)	0.0792 (14)
H1A	2.0022	0.5083	0.1558	0.119*
H1B	1.9835	0.5118	0.3005	0.119*
H1C	1.7765	0.5485	0.2141	0.119*
C2	1.6709 (7)	0.46177 (17)	0.2185 (4)	0.0576 (11)
C3	1.5819 (8)	0.43632 (17)	0.1076 (4)	0.0586 (11)
H3	1.6539	0.4500	0.0343	0.070*
C4	1.3897 (7)	0.39125 (16)	0.1021 (3)	0.0498 (9)
H4	1.3321	0.3754	0.0256	0.060*
C5	1.2814 (7)	0.36934 (15)	0.2103 (3)	0.0409 (8)
C6	1.3763 (8)	0.39378 (17)	0.3217 (3)	0.0498 (9)
H6	1.3103	0.3794	0.3956	0.060*
C7	1.5673 (8)	0.43920 (17)	0.3253 (4)	0.0623 (11)
H7	1.6273	0.4549	0.4017	0.075*
C8	0.9566 (7)	0.30543 (15)	0.1167 (3)	0.0436 (8)
H8	0.9950	0.3238	0.0424	0.052*
C9	0.7549 (7)	0.25849 (15)	0.1140 (3)	0.0407 (8)
C10	0.6197 (7)	0.23554 (16)	0.0104 (3)	0.0501 (10)
H10	0.6498	0.2486	−0.0696	0.060*
C11	0.4313 (7)	0.19048 (16)	0.0363 (3)	0.0479 (9)
H11	0.3243	0.1699	−0.0236	0.057*
C12	0.4260 (7)	0.18089 (14)	0.1598 (3)	0.0379 (8)
N1	1.0843 (6)	0.32285 (12)	0.2158 (2)	0.0413 (7)
N2	0.2502 (6)	0.13966 (14)	0.2189 (3)	0.0488 (8)
O1	0.2643 (6)	0.13733 (12)	0.3329 (2)	0.0663 (8)
O2	0.0953 (6)	0.10886 (13)	0.1531 (3)	0.0692 (8)
S1	0.64676 (18)	0.22535 (4)	0.24745 (7)	0.0417 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.058 (3)	0.047 (2)	0.133 (4)	−0.009 (2)	0.009 (3)	−0.004 (3)
C2	0.036 (2)	0.042 (2)	0.095 (3)	0.0044 (17)	0.008 (2)	0.003 (2)
C3	0.052 (2)	0.050 (2)	0.074 (3)	0.000 (2)	0.010 (2)	0.013 (2)
C4	0.049 (2)	0.051 (2)	0.050 (2)	0.0003 (19)	0.0058 (17)	0.0034 (18)
C5	0.0348 (19)	0.0387 (19)	0.0492 (19)	0.0028 (15)	0.0013 (15)	0.0042 (16)
C6	0.048 (2)	0.052 (2)	0.049 (2)	−0.0071 (19)	−0.0010 (16)	0.0026 (18)

C7	0.055 (2)	0.058 (3)	0.073 (3)	−0.008 (2)	−0.005 (2)	−0.008 (2)
C8	0.042 (2)	0.050 (2)	0.0386 (17)	0.0005 (17)	0.0031 (15)	0.0093 (16)
C9	0.041 (2)	0.044 (2)	0.0360 (17)	0.0019 (16)	−0.0023 (14)	0.0015 (15)
C10	0.051 (2)	0.065 (2)	0.0339 (17)	−0.006 (2)	−0.0021 (16)	0.0042 (17)
C11	0.049 (2)	0.058 (2)	0.0354 (17)	−0.0040 (19)	−0.0052 (15)	−0.0093 (17)
C12	0.0331 (18)	0.0394 (19)	0.0413 (16)	0.0046 (15)	0.0039 (14)	−0.0025 (15)
N1	0.0417 (16)	0.0423 (16)	0.0393 (14)	0.0015 (14)	−0.0032 (12)	0.0033 (13)
N2	0.0507 (19)	0.0464 (19)	0.0493 (17)	0.0056 (16)	0.0041 (15)	0.0033 (15)
O1	0.085 (2)	0.0671 (18)	0.0480 (15)	−0.0064 (16)	0.0113 (14)	0.0092 (14)
O2	0.0684 (19)	0.0685 (18)	0.0704 (17)	−0.0220 (16)	0.0005 (15)	−0.0036 (16)
S1	0.0472 (6)	0.0464 (6)	0.0313 (4)	0.0021 (4)	−0.0015 (4)	−0.0003 (4)

Geometric parameters (Å, º)

C1—C2	1.513 (5)	C7—H7	0.9300
C1—H1A	0.9600	C8—N1	1.269 (4)
C1—H1B	0.9600	C8—C9	1.439 (5)
C1—H1C	0.9600	C8—H8	0.9300
C2—C7	1.374 (5)	C9—C10	1.366 (4)
C2—C3	1.378 (5)	C9—S1	1.725 (3)
C3—C4	1.377 (5)	C10—C11	1.402 (4)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.391 (4)	C11—C12	1.351 (4)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.381 (5)	C12—N2	1.430 (4)
C5—N1	1.420 (4)	C12—S1	1.713 (3)
C6—C7	1.379 (5)	N2—O2	1.222 (4)
C6—H6	0.9300	N2—O1	1.228 (4)
C2—C1—H1A	109.5	C2—C7—H7	119.3
C2—C1—H1B	109.5	C6—C7—H7	119.3
H1A—C1—H1B	109.5	N1—C8—C9	123.0 (3)
C2—C1—H1C	109.5	N1—C8—H8	118.5
H1A—C1—H1C	109.5	C9—C8—H8	118.5
H1B—C1—H1C	109.5	C10—C9—C8	126.2 (3)
C7—C2—C3	117.4 (4)	C10—C9—S1	111.4 (3)
C7—C2—C1	121.3 (4)	C8—C9—S1	122.4 (2)
C3—C2—C1	121.3 (4)	C9—C10—C11	113.6 (3)
C4—C3—C2	122.0 (4)	C9—C10—H10	123.2
C4—C3—H3	119.0	C11—C10—H10	123.2
C2—C3—H3	119.0	C12—C11—C10	110.9 (3)
C3—C4—C5	120.3 (4)	C12—C11—H11	124.6
C3—C4—H4	119.8	C10—C11—H11	124.6
C5—C4—H4	119.8	C11—C12—N2	125.8 (3)
C6—C5—C4	117.7 (3)	C11—C12—S1	114.1 (3)
C6—C5—N1	117.1 (3)	N2—C12—S1	120.1 (2)
C4—C5—N1	125.2 (3)	C8—N1—C5	119.4 (3)
C7—C6—C5	121.1 (3)	O2—N2—O1	124.0 (3)
C7—C6—H6	119.4	O2—N2—C12	118.1 (3)
C5—C6—H6	119.4	O1—N2—C12	118.0 (3)

C2—C7—C6	121.4 (4)	C12—S1—C9	89.98 (15)
C7—C2—C3—C4	2.1 (6)	C9—C10—C11—C12	0.8 (4)
C1—C2—C3—C4	−178.5 (3)	C10—C11—C12—N2	177.6 (3)
C2—C3—C4—C5	−0.9 (6)	C10—C11—C12—S1	−0.2 (4)
C3—C4—C5—C6	−0.9 (5)	C9—C8—N1—C5	179.7 (3)
C3—C4—C5—N1	−179.5 (3)	C6—C5—N1—C8	168.5 (3)
C4—C5—C6—C7	1.4 (5)	C4—C5—N1—C8	−12.8 (5)
N1—C5—C6—C7	−179.9 (3)	C11—C12—N2—O2	2.2 (5)
C3—C2—C7—C6	−1.6 (6)	S1—C12—N2—O2	179.8 (3)
C1—C2—C7—C6	179.1 (4)	C11—C12—N2—O1	−177.9 (3)
C5—C6—C7—C2	−0.2 (6)	S1—C12—N2—O1	−0.2 (4)
N1—C8—C9—C10	−176.6 (3)	C11—C12—S1—C9	−0.3 (3)
N1—C8—C9—S1	5.4 (5)	N2—C12—S1—C9	−178.2 (3)
C8—C9—C10—C11	−179.2 (3)	C10—C9—S1—C12	0.7 (3)
S1—C9—C10—C11	−1.0 (4)	C8—C9—S1—C12	179.0 (3)