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Thiophene-2-carbaldehyde 2,4-dinitrophenylhydrazone

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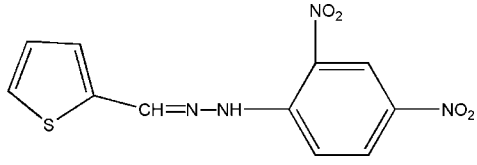
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.087; data-to-parameter ratio = 12.4.

In the approximately planar molecule of the title compound, $C_{11}H_8N_4O_4S$, the dihedral angle between the thiophene and benzene rings is 5.73 (10)°. In the crystal structure, bifurcated inter/intramolecular N—H···(O,O) hydrogen bonds are present. The intermolecular links lead to inversion dimers containing an $R_2^2(12)$ graph-set motif.

Related literature

For general background, see: Okabe *et al.* (1993). For graph-set notation, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{11}H_8N_4O_4S$
M_r = 292.27
Monoclinic, $P2_1/c$
a = 4.8994 (17) Å
b = 9.520 (3) Å
c = 25.708 (8) Å
β = 92.71 (2)°
V = 1197.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 291 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.915, T_{max} = 0.929
11010 measured reflections
2285 independent reflections
1718 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.087
S = 1.02
2285 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1···O1^{i}	0.87 (2)	2.57 (2)	3.338 (2)	148.2 (19)
N3—H1···O1	0.87 (2)	2.01 (2)	2.630 (2)	127.4 (19)

Symmetry code: (i) $-x+1, -y+1, -z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2406).

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supporting information

Acta Cryst. (2008). E**64**, o2421 [doi:10.1107/S1600536808038397]

Thiophene-2-carbaldehyde 2,4-dinitrophenylhydrazone

Zhi-gang Yin, Heng-yu Qian, He-ping Li, Jie Hu and Chun-xia Zhang

S1. Comment

2,4-Dinitrophenylhydrazine is a reagent which is widely used for condensation with aldehydes and ketones. Several phenylhydrazone derivatives have been shown to be potentially DNA-damaging and are mutagenic agents(Okabe *et al.* 1993). As part of our work, we have synthesized the title compound (I) and reported its cyrstal structure.

The title molecule is roughly planar. Indeed, the benzene and the thiophene rings are only slight twisted, making a dihedral angle of 5.73 (10)° (Fig. 1). The two nitro groups, O1/N1/O2 and O3/N2/O4 are coplanar with the benzene ring to which they are attached.

Intermolecular N-H···O hydrogen bonds link the molecule two by two around inversion center, building then a $R_2^2(12)$ graph set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995) if the intramolecular N-H···O hydrogen bonds are ignored (Table 1, Fig.2).

S2. Experimental

2,4-dinitrophenylhydrazine(1 mmol, 0.198 g) was dissolved in anhydrous methanol, H_2SO_4 (98% 0.5 ml) was added to this, the mixture was stirred for several minitutes at 351 K, thiophene-2-carbaldehyde (1 mmol 0.112 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in methanol, purple single crystals of (I) was obtained after 5 d.

S3. Refinement

All H atoms were placed in calculated positions and treated as riding with C—H=0.93Å (aromatic) and N—H=0.86Å with U_{iso}(H)=1.2U_{eq}(C or N).

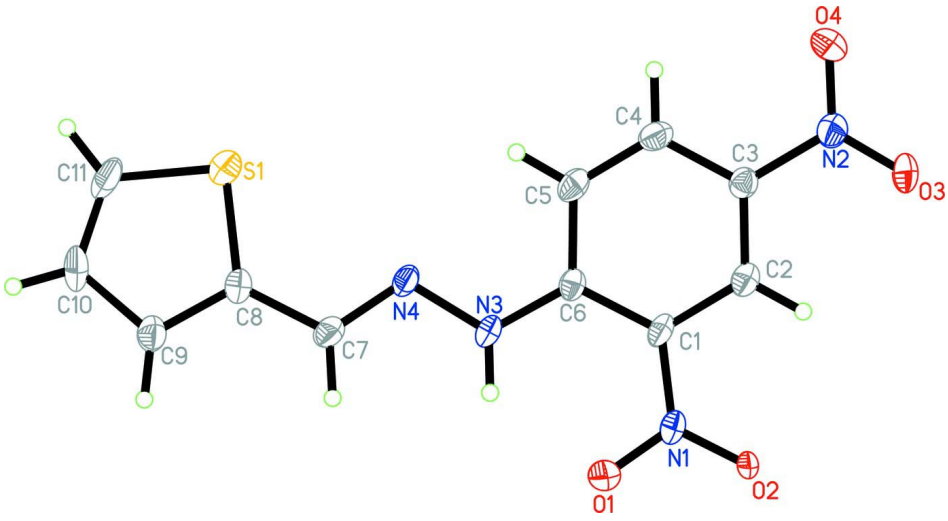

Figure 1

Molecular view of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

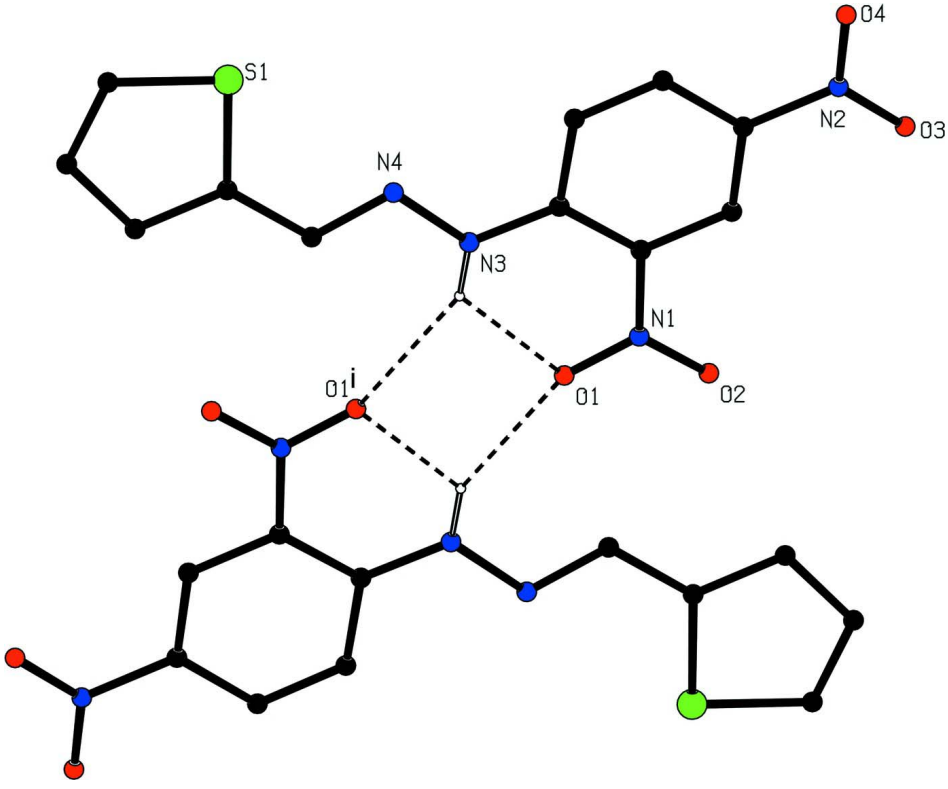

Figure 2

View showing the N-H···O hydrogen bonds buiding a $R_2^2(12)$ graph set motif. Hydrogen bonds are shown as dashed lines.

Thiophene-2-carbaldehyde 2,4-dinitrophenylhydrazone

Crystal data

$C_{11}H_8N_4O_4S$
$M_r = 292.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.8994$ (17) Å
$b = 9.520$ (3) Å
$c = 25.708$ (8) Å
$\beta = 92.71$ (2)°
$V = 1197.7$ (7) Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.621$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5391 reflections
$\theta = 2.3$–$27.4°$
$\mu = 0.29$ mm^{-1}
$T = 291$ K
Block, purple
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.915$, $T_{max} = 0.929$
11010 measured reflections
2285 independent reflections
1718 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.6°$
$h = -6 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -31 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.087$
$S = 1.02$
2285 reflections
184 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1154 (4)	0.2360 (2)	0.04553 (7)	0.0342 (4)
C2	−0.0759 (4)	0.1287 (2)	0.04720 (7)	0.0350 (4)
H2	−0.2010	0.1150	0.0193	0.042*

C3	−0.0801 (4)	0.04351 (18)	0.08984 (7)	0.0315 (4)
C4	0.1065 (4)	0.0673 (2)	0.13207 (7)	0.0383 (5)
H4	0.1048	0.0087	0.1610	0.046*
C5	0.2882 (4)	0.1738 (2)	0.13141 (7)	0.0350 (4)
H5	0.4096	0.1870	0.1599	0.042*
C6	0.2980 (4)	0.26512 (18)	0.08856 (7)	0.0291 (4)
C7	0.8165 (4)	0.49823 (19)	0.13079 (7)	0.0389 (5)
H7	0.7974	0.5614	0.1033	0.047*
C8	1.0163 (4)	0.52284 (19)	0.17283 (7)	0.0341 (4)
C9	1.1994 (5)	0.6353 (2)	0.17481 (8)	0.0418 (5)
H9	1.2066	0.7050	0.1496	0.050*
C10	1.3738 (5)	0.6289 (2)	0.22076 (9)	0.0465 (5)
H10	1.5107	0.6939	0.2290	0.056*
C11	1.3173 (5)	0.5174 (2)	0.25090 (8)	0.0522 (6)
H11	1.4113	0.4975	0.2823	0.063*
N1	0.0973 (4)	0.31477 (18)	0.00000 (6)	0.0394 (4)
N2	−0.2778 (4)	−0.06932 (17)	0.09065 (7)	0.0414 (4)
N3	0.4846 (4)	0.37052 (17)	0.08923 (6)	0.0378 (4)
H1	0.483 (5)	0.426 (2)	0.0624 (9)	0.045*
N4	0.6631 (3)	0.38663 (15)	0.13180 (6)	0.0321 (4)
O1	0.2484 (4)	0.41150 (16)	−0.00353 (6)	0.0545 (4)
O2	−0.0594 (3)	0.29004 (14)	−0.03604 (5)	0.0461 (4)
O3	−0.4272 (3)	−0.08987 (16)	0.05310 (6)	0.0526 (4)
O4	−0.2847 (3)	−0.13710 (15)	0.12986 (6)	0.0513 (4)
S1	1.05706 (12)	0.41736 (6)	0.22559 (2)	0.04762 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0302 (10)	0.0438 (10)	0.0277 (10)	−0.0016 (8)	−0.0096 (8)	−0.0034 (7)
C2	0.0330 (10)	0.0410 (10)	0.0303 (10)	−0.0007 (8)	−0.0046 (8)	−0.0039 (7)
C3	0.0326 (10)	0.0290 (8)	0.0325 (9)	−0.0001 (7)	−0.0024 (8)	−0.0025 (7)
C4	0.0414 (12)	0.0417 (10)	0.0314 (10)	0.0038 (8)	−0.0038 (9)	0.0075 (8)
C5	0.0259 (10)	0.0463 (10)	0.0323 (10)	0.0090 (8)	−0.0035 (8)	−0.0005 (8)
C6	0.0270 (9)	0.0339 (8)	0.0262 (9)	0.0002 (7)	−0.0004 (7)	−0.0062 (7)
C7	0.0510 (12)	0.0394 (10)	0.0257 (9)	−0.0026 (9)	−0.0057 (8)	−0.0013 (8)
C8	0.0316 (10)	0.0353 (9)	0.0349 (9)	−0.0023 (8)	−0.0025 (8)	−0.0079 (7)
C9	0.0473 (13)	0.0361 (9)	0.0412 (11)	−0.0042 (9)	−0.0073 (9)	−0.0059 (8)
C10	0.0387 (12)	0.0416 (10)	0.0581 (14)	−0.0045 (9)	−0.0105 (10)	−0.0220 (10)
C11	0.0629 (15)	0.0558 (13)	0.0357 (11)	0.0052 (11)	−0.0222 (11)	−0.0131 (9)
N1	0.0383 (10)	0.0452 (9)	0.0335 (9)	−0.0104 (8)	−0.0120 (8)	−0.0013 (7)
N2	0.0360 (10)	0.0421 (9)	0.0453 (10)	−0.0026 (7)	−0.0074 (8)	0.0047 (8)
N3	0.0387 (10)	0.0443 (9)	0.0290 (9)	−0.0077 (7)	−0.0126 (7)	−0.0001 (7)
N4	0.0345 (8)	0.0344 (8)	0.0266 (8)	−0.0003 (6)	−0.0087 (7)	−0.0040 (6)
O1	0.0659 (11)	0.0525 (9)	0.0435 (9)	−0.0225 (8)	−0.0163 (8)	0.0158 (7)
O2	0.0471 (9)	0.0505 (8)	0.0383 (8)	−0.0317 (7)	−0.0222 (7)	0.0182 (6)
O3	0.0488 (10)	0.0534 (9)	0.0537 (9)	−0.0260 (7)	−0.0185 (8)	−0.0002 (7)
O4	0.0566 (10)	0.0451 (8)	0.0519 (9)	−0.0169 (7)	−0.0008 (8)	0.0131 (7)

S1	0.0480 (3)	0.0553 (3)	0.0385 (3)	−0.0071 (2)	−0.0093 (2)	0.0040 (2)

Geometric parameters (Å, º)

C1—C2	1.388 (3)	C8—C9	1.396 (3)
C1—N1	1.389 (2)	C8—S1	1.692 (2)
C1—C6	1.417 (2)	C9—C10	1.426 (3)
C2—C3	1.365 (3)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.351 (3)
C3—C4	1.404 (2)	C10—H10	0.9300
C3—N2	1.447 (2)	C11—S1	1.696 (2)
C4—C5	1.350 (3)	C11—H11	0.9300
C4—H4	0.9300	N1—O1	1.188 (2)
C5—C6	1.406 (3)	N1—O2	1.1983 (19)
C5—H5	0.9300	N2—O4	1.199 (2)
C6—N3	1.357 (2)	N2—O3	1.199 (2)
C7—N4	1.302 (2)	N3—N4	1.377 (2)
C7—C8	1.442 (2)	N3—H1	0.87 (2)
C7—H7	0.9300		
C2—C1—N1	114.01 (15)	C9—C8—S1	112.01 (14)
C2—C1—C6	121.47 (17)	C7—C8—S1	123.66 (14)
N1—C1—C6	124.40 (17)	C8—C9—C10	110.86 (19)
C3—C2—C1	119.87 (17)	C8—C9—H9	124.6
C3—C2—H2	120.1	C10—C9—H9	124.6
C1—C2—H2	120.1	C11—C10—C9	112.19 (18)
C2—C3—C4	119.41 (17)	C11—C10—H10	123.9
C2—C3—N2	119.22 (15)	C9—C10—H10	123.9
C4—C3—N2	121.36 (16)	C10—C11—S1	113.08 (15)
C5—C4—C3	121.14 (18)	C10—C11—H11	123.5
C5—C4—H4	119.4	S1—C11—H11	123.5
C3—C4—H4	119.4	O1—N1—O2	118.16 (16)
C4—C5—C6	121.43 (17)	O1—N1—C1	117.89 (14)
C4—C5—H5	119.3	O2—N1—C1	123.94 (16)
C6—C5—H5	119.3	O4—N2—O3	123.26 (17)
N3—C6—C5	119.70 (15)	O4—N2—C3	117.21 (15)
N3—C6—C1	123.69 (17)	O3—N2—C3	119.53 (16)
C5—C6—C1	116.55 (17)	C6—N3—N4	119.65 (16)
N4—C7—C8	119.31 (16)	C6—N3—H1	117.6 (14)
N4—C7—H7	120.3	N4—N3—H1	122.8 (14)
C8—C7—H7	120.3	C7—N4—N3	114.87 (15)
C9—C8—C7	124.33 (18)	C8—S1—C11	91.85 (11)
N1—C1—C2—C3	180.00 (18)	C8—C9—C10—C11	−0.6 (3)
C6—C1—C2—C3	−3.8 (3)	C9—C10—C11—S1	0.1 (3)
C1—C2—C3—C4	1.3 (3)	C2—C1—N1—O1	177.51 (19)
C1—C2—C3—N2	−178.99 (18)	C6—C1—N1—O1	1.5 (3)
C2—C3—C4—C5	0.6 (3)	C2—C1—N1—O2	−3.3 (3)

N2—C3—C4—C5	−179.16 (18)	C6—C1—N1—O2	−179.4 (2)
C3—C4—C5—C6	0.1 (3)	C2—C3—N2—O4	−176.10 (19)
C4—C5—C6—N3	−179.66 (19)	C4—C3—N2—O4	3.6 (3)
C4—C5—C6—C1	−2.5 (3)	C2—C3—N2—O3	3.1 (3)
C2—C1—C6—N3	−178.60 (18)	C4—C3—N2—O3	−177.21 (19)
N1—C1—C6—N3	−2.8 (3)	C5—C6—N3—N4	0.2 (3)
C2—C1—C6—C5	4.3 (3)	C1—C6—N3—N4	−176.76 (17)
N1—C1—C6—C5	−179.88 (19)	C8—C7—N4—N3	−178.20 (17)
N4—C7—C8—C9	177.13 (19)	C6—N3—N4—C7	−173.88 (18)
N4—C7—C8—S1	−2.5 (3)	C9—C8—S1—C11	−0.66 (18)
C7—C8—C9—C10	−178.8 (2)	C7—C8—S1—C11	179.01 (19)
S1—C8—C9—C10	0.8 (2)	C10—C11—S1—C8	0.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H1···O1^{i}	0.87 (2)	2.57 (2)	3.338 (2)	148.2 (19)
N3—H1···O1	0.87 (2)	2.01 (2)	2.630 (2)	127.4 (19)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.