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1-[1-(4-Nitrophenyl)ethylidene]thiosemicarbazide

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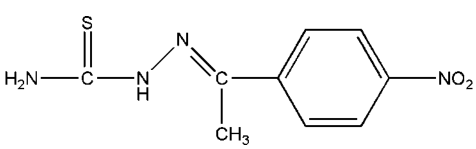
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.141; data-to-parameter ratio = 15.9.

The title compound, $C_9H_{10}N_4O_2S$, was prepared by the reaction of 1-(4-nitrophenyl)ethanone and thiosemicarbazide in ethanol at 367 K. There are weak intermolecular N—H⋯S and N—H⋯O hydrogen-bonding interactions in the crystal structure involving the amine and nitrile groups, respectively, as donors.

Related literature

For related literature, see: Jian *et al.* (2006); Qin *et al.* (2006); Rozwadowski *et al.* (1999).



Experimental

Crystal data

$C_9H_{10}N_4O_2S$
M_r = 238.27
Triclinic, $P\overline{1}$
a = 7.4450 (15) Å
b = 9.3180 (19) Å
c = 9.4050 (19) Å
α = 62.08 (3)°
β = 76.41 (3)°
γ = 69.02 (3)°
V = 536.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 (2) K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
2493 measured reflections
2307 independent reflections
1776 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.140
S = 1.08
2307 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3A⋯S1^{i}	0.86	2.74	3.581 (2)	166
N4—H4A⋯O1^{ii}	0.86	2.35	3.101 (3)	146
N4—H4B⋯O2^{iii}	0.86	2.29	3.133 (3)	166

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y, -z+2$; (iii) $x-1, y+1, z-1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2598).

References

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supplementary materials

Acta Cryst. (2008). E**64**, o1730 [doi:10.1107/S1600536808025105]

1-[1-(4-Nitrophenyl)ethylidene]thiosemicarbazide

J.-G. Wang, F.-F. Jian and Y.-F. Ding

Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry (Jian *et al.*, 2006). Schiff bases show biochemical and pharmacological applications. The growing interest in Schiff bases lately is also due to their ability to form intramolecular hydrogen bonds by electron coupling between acid-base centers (Rozwadowski *et al.*,1999). The title compound (I), was synthesized and we report here its crystal structure

In the crystal structure of (I) (Fig. 1). The C6–C9/N2/N3/S1 plane makes a dihedral angle of 19.78 (127)° with the benzene ring (C1—C6). The C═N bond length [1.281 (3) Å] and C═S bond length [1.685 (2) Å] are in agreement with those observed before (Jian *et al.*, 2006; Qin *et al.*, 2006). There are intermolecular N–H···S and N–H···O hydrogen-bond interactions to stabilize the crystal structure (Table 1).

Experimental

A mixture of hydrochloric acid 0.6 mL (0.02 mol) and thiosemicarbazide 1.8 g (0.02 mol) was stirred with ethanol (50 mL) at 293 K for 2 h, then add 1-(4-nitrophenyl)ethanone 3.3 g (0.02 mol), then afford the title compound [4.17 g, yield: 87.6%]. Single crystals suitable for X-ray measurements were obtained by recrystallization from acetone and ethanol(1:1) at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H and N—H distances of 0.93–0.96 and 0.86 Å, and with U_{iso}=1.2 or 1.5U_{eq}.

Figures

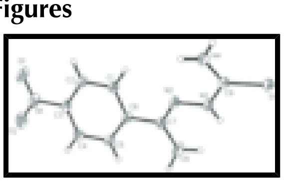

Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

1-[1-(4-Nitrophenyl)ethylidene]thiosemicarbazide

Crystal data

$C_9H_{10}N_4O_2S$	$Z = 2$
$M_r = 238.27$	$F_{000} = 248$
Triclinic, $P\bar{1}$	$D_x = 1.475$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.4450 (15) Å	Cell parameters from 1776 reflections
b = 9.3180 (19) Å	θ = 2.5–27.0°
c = 9.4050 (19) Å	μ = 0.29 mm^{-1}
α = 62.08 (3)°	T = 293 (2) K
β = 76.41 (3)°	Block, yellow
γ = 69.02 (3)°	0.20 × 0.15 × 0.10 mm
V = 536.5 (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1776 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.026
Monochromator: graphite	θ_{max} = 27.0°
T = 293(2) K	θ_{min} = 2.5°
φ and ω scans	h = 0→8
Absorption correction: none	k = −11→11
2493 measured reflections	l = −11→11
2307 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.140	$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 0.1605P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ < 0.001
2307 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
S1	−0.18728 (10)	0.97254 (7)	0.38617 (7)	0.0483 (2)
O1	0.2761 (3)	−0.3046 (2)	1.2287 (3)	0.0697 (6)
O2	0.4183 (3)	−0.2729 (2)	1.3830 (2)	0.0680 (6)
N1	0.3337 (3)	−0.2167 (2)	1.2626 (2)	0.0472 (5)
N2	0.0574 (3)	0.5445 (2)	0.7336 (2)	0.0373 (4)
N3	0.0128 (3)	0.7117 (2)	0.6222 (2)	0.0394 (4)
H3A	0.0744	0.7778	0.6144	0.047*
N4	−0.2206 (3)	0.6599 (2)	0.5464 (2)	0.0521 (5)
H4A	−0.1876	0.5573	0.6190	0.063*
H4B	−0.3121	0.6918	0.4873	0.063*
C1	0.1766 (3)	0.1958 (3)	0.9090 (3)	0.0392 (5)
H1A	0.1182	0.2381	0.8145	0.047*
C2	0.2135 (3)	0.0244 (3)	1.0120 (3)	0.0410 (5)
H2B	0.1813	−0.0485	0.9871	0.049*
C3	0.2990 (3)	−0.0353 (2)	1.1521 (3)	0.0364 (5)
C4	0.3527 (3)	0.0684 (3)	1.1924 (3)	0.0415 (5)
H4C	0.4109	0.0249	1.2873	0.050*
C5	0.3167 (3)	0.2400 (3)	1.0864 (3)	0.0397 (5)
H5A	0.3537	0.3112	1.1100	0.048*
C6	0.2260 (3)	0.3065 (2)	0.9453 (2)	0.0337 (4)
C7	0.1848 (3)	0.4909 (2)	0.8317 (3)	0.0371 (5)
C8	0.2907 (4)	0.5977 (3)	0.8389 (4)	0.0631 (8)
H8A	0.2478	0.7120	0.7589	0.095*
H8B	0.4268	0.5521	0.8185	0.095*
H8C	0.2649	0.5972	0.9441	0.095*
C9	−0.1299 (3)	0.7696 (3)	0.5255 (3)	0.0384 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0686 (4)	0.0278 (3)	0.0474 (4)	−0.0157 (3)	−0.0254 (3)	−0.0044 (2)
O1	0.0890 (15)	0.0312 (9)	0.0844 (14)	−0.0239 (9)	−0.0267 (11)	−0.0073 (9)
O2	0.0795 (14)	0.0444 (10)	0.0588 (11)	−0.0168 (9)	−0.0295 (10)	0.0051 (9)
N1	0.0442 (11)	0.0309 (9)	0.0534 (12)	−0.0102 (8)	−0.0077 (9)	−0.0062 (8)
N2	0.0450 (10)	0.0227 (8)	0.0400 (9)	−0.0076 (7)	−0.0105 (8)	−0.0084 (7)
N3	0.0478 (10)	0.0242 (8)	0.0446 (10)	−0.0109 (7)	−0.0151 (8)	−0.0078 (7)
N4	0.0678 (14)	0.0332 (10)	0.0558 (12)	−0.0205 (9)	−0.0276 (10)	−0.0047 (9)
C1	0.0448 (12)	0.0296 (10)	0.0433 (11)	−0.0090 (8)	−0.0139 (9)	−0.0120 (9)
C2	0.0468 (12)	0.0285 (10)	0.0513 (13)	−0.0119 (9)	−0.0106 (10)	−0.0158 (9)
C3	0.0352 (11)	0.0254 (9)	0.0404 (11)	−0.0070 (8)	−0.0027 (8)	−0.0088 (8)
C4	0.0462 (12)	0.0361 (11)	0.0392 (11)	−0.0105 (9)	−0.0112 (9)	−0.0109 (9)
C5	0.0481 (12)	0.0296 (10)	0.0444 (12)	−0.0100 (9)	−0.0119 (9)	−0.0152 (9)
C6	0.0347 (10)	0.0242 (9)	0.0400 (11)	−0.0063 (8)	−0.0064 (8)	−0.0119 (8)
C7	0.0404 (11)	0.0249 (9)	0.0442 (11)	−0.0068 (8)	−0.0093 (9)	−0.0124 (9)

C8	0.0745 (18)	0.0316 (11)	0.0821 (19)	−0.0198 (12)	−0.0407 (15)	−0.0044 (12)
C9	0.0481 (12)	0.0284 (10)	0.0379 (11)	−0.0111 (9)	−0.0093 (9)	−0.0108 (8)

Geometric parameters (Å, °)

S1—C9	1.685 (2)	C1—H1A	0.9300
O1—N1	1.231 (3)	C2—C3	1.381 (3)
O2—N1	1.224 (3)	C2—H2B	0.9300
N1—C3	1.473 (3)	C3—C4	1.389 (3)
N2—C7	1.281 (3)	C4—C5	1.395 (3)
N2—N3	1.379 (2)	C4—H4C	0.9300
N3—C9	1.353 (3)	C5—C6	1.397 (3)
N3—H3A	0.8600	C5—H5A	0.9300
N4—C9	1.336 (3)	C6—C7	1.498 (3)
N4—H4A	0.8600	C7—C8	1.506 (3)
N4—H4B	0.8600	C8—H8A	0.9600
C1—C2	1.388 (3)	C8—H8B	0.9600
C1—C6	1.406 (3)	C8—H8C	0.9600
O2—N1—O1	123.1 (2)	C3—C4—H4C	121.0
O2—N1—C3	118.7 (2)	C5—C4—H4C	121.0
O1—N1—C3	118.15 (19)	C4—C5—C6	121.2 (2)
C7—N2—N3	119.08 (18)	C4—C5—H5A	119.4
C9—N3—N2	118.64 (17)	C6—C5—H5A	119.4
C9—N3—H3A	120.7	C5—C6—C1	118.60 (18)
N2—N3—H3A	120.7	C5—C6—C7	121.53 (18)
C9—N4—H4A	120.0	C1—C6—C7	119.86 (18)
C9—N4—H4B	120.0	N2—C7—C6	114.93 (18)
H4A—N4—H4B	120.0	N2—C7—C8	125.16 (19)
C2—C1—C6	121.03 (19)	C6—C7—C8	119.91 (18)
C2—C1—H1A	119.5	C7—C8—H8A	109.5
C6—C1—H1A	119.5	C7—C8—H8B	109.5
C3—C2—C1	118.5 (2)	H8A—C8—H8B	109.5
C3—C2—H2B	120.8	C7—C8—H8C	109.5
C1—C2—H2B	120.8	H8A—C8—H8C	109.5
C2—C3—C4	122.67 (19)	H8B—C8—H8C	109.5
C2—C3—N1	118.13 (19)	N4—C9—N3	117.19 (18)
C4—C3—N1	119.20 (19)	N4—C9—S1	122.63 (17)
C3—C4—C5	118.0 (2)	N3—C9—S1	120.19 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···S1^{i}	0.86	2.74	3.581 (2)	166
N4—H4A···O1^{ii}	0.86	2.35	3.101 (3)	146
N4—H4B···O2^{iii}	0.86	2.29	3.133 (3)	166

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*, −*y*, −*z*+2; (iii) *x*−1, *y*+1, *z*−1.

Fig. 1