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Benzaldehyde thiosemicarbazone

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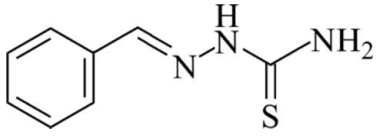
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.058; wR factor = 0.162; data-to-parameter ratio = 14.4.

The title compound, $C_8H_9N_3S$, contains two molecules in the asymmetric unit. One molecule is close to being planar (r.m.s. deviation from the mean plane = 0.06 Å for the non-H atoms), while the other exhibits a dihedral angle of 21.7 (1)° between the benzene ring and the mean plane of the thiosemicarbazone unit. Intermolecular N—H···S hydrogen bonds link the molecules into layers parallel to the (010) plane.

Related literature

For background literature concerning arylhydrazone compounds, see: Beraldo & Gambino (2004); Bondock *et al.* (2007). For the related 2,4-dichlorobenzylidene compound, see: Jing *et al.* (2006).



Experimental

Crystal data

$C_8H_9N_3S$
M_r = 179.24
Triclinic, $P\overline{1}$
a = 5.8692 (13) Å
b = 12.513 (2) Å
c = 13.519 (2) Å
α = 112.735 (3)°
β = 95.384 (2)°
γ = 96.153 (2)°
V = 900.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 298 (2) K
0.24 × 0.13 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.930, T_{max} = 0.970
4740 measured reflections
3124 independent reflections
1846 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.162
S = 0.95
3124 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.86	2.64	3.443 (3)	155
N3—H3A···S1^{ii}	0.86	2.98	3.488 (3)	120
N5—H5···S1^{ii}	0.86	2.61	3.441 (3)	162
N6—H6B···S2^{iii}	0.86	2.51	3.368 (3)	173

Symmetry codes: (i) $-x+3, -y+2, -z+2$; (ii) $x-1, y, z$; (iii) $-x+2, -y+2, -z+1$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2310).

References

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supporting information

Acta Cryst. (2008). E**64**, o2412 [doi:10.1107/S1600536808038270]

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S1. Comment

Aryl-hydrazones, such as semicarbazones, thiosemicarbazones and guanyl hydrazones, often exhibit strong biological activity and are important compounds for drug design (Beraldo & Gambino, 2004), organocatalysis and the preparation of heterocyclic rings (Bondock *et al.*, 2007).

S2. Experimental

Benzaldehyde (0.3 mmol), thiosemicarbazide (0.3 mmol) and 10 ml water were mixed in a 50 ml flask. After stirring for 30 min at 373 K, the resulting mixture was recrystallized from ethanol, affording the title compound as colourless crystals. Elemental analysis: calculated C 53.61, H 5.06, N 23.44%; found: C 53.58, H 5.55, N 23.51%.

S3. Refinement

H atoms were placed in geometrically idealized positions (N—H = 0.86, C—H = 0.93 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C/N).

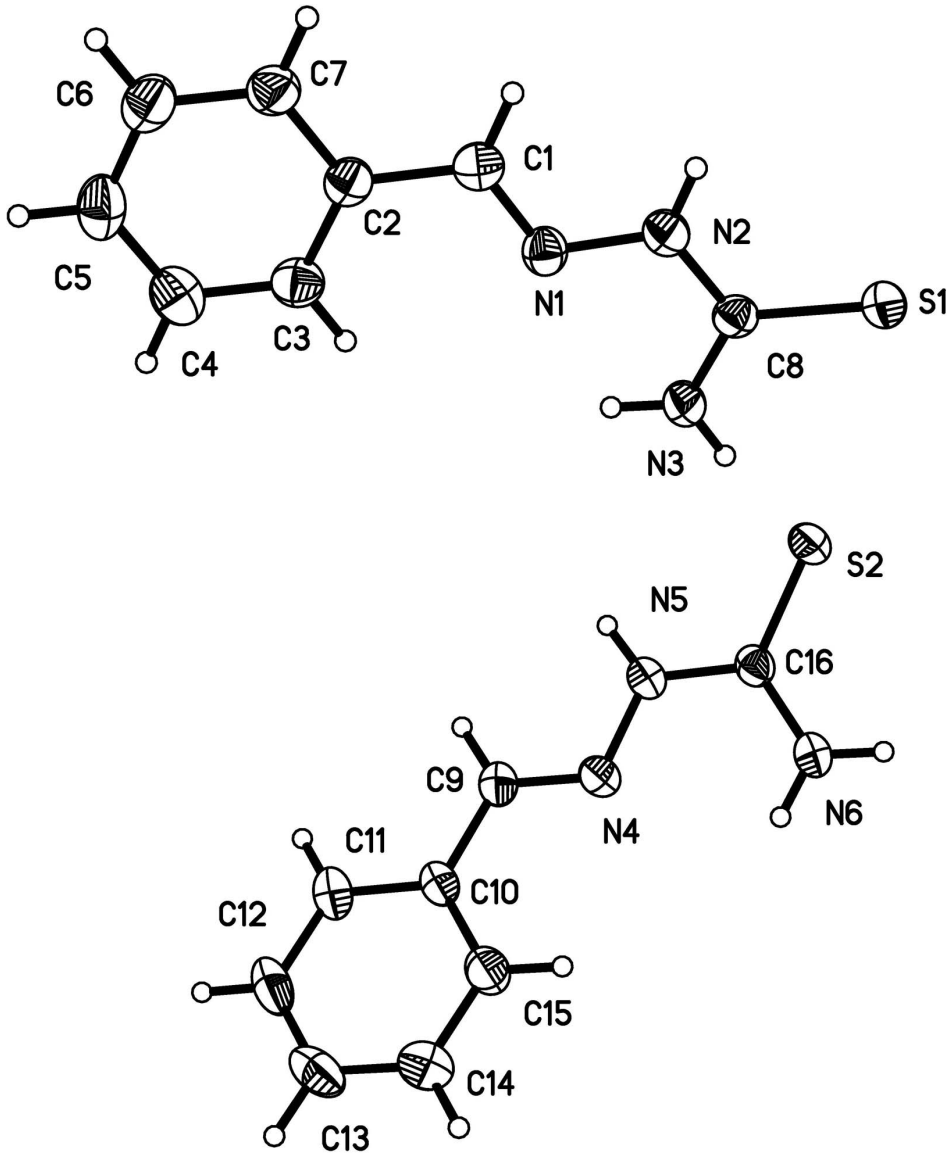

Figure 1

Two molecules in the asymmetric unit of the title compound with displacement ellipsoids shown at 30% probability for non-H atoms.

Benzaldehyde thiosemicarbazone

Crystal data

$C_8H_9N_3S$
$M_r = 179.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.8692\ (13)$ Å
$b = 12.513\ (2)$ Å
$c = 13.519\ (2)$ Å
$\alpha = 112.735\ (3)°$
$\beta = 95.384\ (2)°$
$\gamma = 96.153\ (2)°$
$V = 900.4\ (3)$ Å^3
$Z = 4$
$F(000) = 376$
$D_x = 1.322$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1310 reflections
$\theta = 2.9–25.0°$
$\mu = 0.31$ mm^{-1}

T = 298 K
Block, orange

0.24 × 0.13 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.930, T_{max} = 0.970

4740 measured reflections
3124 independent reflections
1846 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −6→6
k = −12→14
l = −16→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.162
S = 0.95
3124 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0856P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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}
N1	0.9674 (5)	0.8260 (3)	0.9684 (2)	0.0484 (8)
N2	1.1667 (5)	0.8822 (3)	0.9512 (2)	0.0504 (8)
H2	1.2934	0.8999	0.9956	0.061*
N3	0.9536 (5)	0.8863 (3)	0.8034 (2)	0.0550 (9)
H3A	0.8331	0.8563	0.8208	0.066*
H3B	0.9414	0.9020	0.7467	0.066*
N4	0.3374 (5)	0.7659 (3)	0.4823 (2)	0.0486 (8)
N5	0.4649 (5)	0.8653 (3)	0.5625 (2)	0.0518 (8)
H5	0.4200	0.8950	0.6249	0.062*
N6	0.7036 (6)	0.8737 (3)	0.4423 (3)	0.0612 (10)
H6A	0.6092	0.8166	0.3930	0.073*
H6B	0.8262	0.9031	0.4259	0.073*
S1	1.40293 (17)	0.96409 (10)	0.83250 (8)	0.0561 (3)
S2	0.83277 (19)	1.02721 (9)	0.64490 (8)	0.0610 (4)

C1	0.9813 (7)	0.8063 (3)	1.0543 (3)	0.0500 (9)
H1	1.1169	0.8341	1.1040	0.060*
C2	0.7837 (7)	0.7398 (3)	1.0749 (3)	0.0493 (10)
C3	0.5747 (7)	0.7039 (3)	1.0076 (3)	0.0569 (10)
H3	0.5566	0.7229	0.9475	0.068*
C4	0.3920 (8)	0.6403 (4)	1.0277 (4)	0.0660 (12)
H4	0.2516	0.6167	0.9814	0.079*
C5	0.4178 (9)	0.6118 (4)	1.1169 (4)	0.0700 (13)
H5A	0.2950	0.5690	1.1309	0.084*
C6	0.6255 (9)	0.6472 (4)	1.1845 (4)	0.0675 (12)
H6	0.6431	0.6283	1.2447	0.081*
C7	0.8081 (8)	0.7104 (3)	1.1640 (3)	0.0584 (11)
H7	0.9486	0.7336	1.2100	0.070*
C8	1.1574 (6)	0.9084 (3)	0.8637 (3)	0.0438 (9)
C9	0.1484 (7)	0.7251 (3)	0.5034 (3)	0.0484 (9)
H9	0.0969	0.7658	0.5687	0.058*
C10	0.0129 (6)	0.6152 (3)	0.4263 (3)	0.0455 (9)
C11	−0.2018 (7)	0.5775 (4)	0.4462 (4)	0.0607 (11)
H11	−0.2622	0.6246	0.5065	0.073*
C12	−0.3267 (8)	0.4700 (4)	0.3766 (4)	0.0695 (13)
H12	−0.4716	0.4453	0.3898	0.083*
C13	−0.2378 (9)	0.4002 (4)	0.2889 (4)	0.0736 (13)
H13	−0.3205	0.3270	0.2435	0.088*
C14	−0.0287 (9)	0.4368 (4)	0.2671 (4)	0.0730 (13)
H14	0.0287	0.3893	0.2060	0.088*
C15	0.0981 (7)	0.5436 (3)	0.3350 (3)	0.0576 (11)
H15	0.2410	0.5680	0.3198	0.069*
C16	0.6604 (6)	0.9162 (3)	0.5434 (3)	0.0443 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0460 (19)	0.0479 (18)	0.0522 (19)	0.0088 (15)	0.0119 (15)	0.0195 (15)
N2	0.0434 (18)	0.056 (2)	0.0511 (19)	0.0056 (15)	0.0037 (15)	0.0225 (16)
N3	0.0415 (19)	0.072 (2)	0.0521 (19)	−0.0021 (16)	0.0018 (16)	0.0296 (17)
N4	0.0452 (19)	0.0479 (18)	0.0471 (18)	−0.0024 (15)	0.0008 (15)	0.0170 (15)
N5	0.048 (2)	0.0513 (19)	0.0488 (19)	−0.0080 (15)	0.0049 (15)	0.0170 (15)
N6	0.055 (2)	0.063 (2)	0.055 (2)	−0.0165 (17)	0.0107 (17)	0.0171 (17)
S1	0.0434 (6)	0.0700 (7)	0.0494 (6)	0.0008 (5)	0.0081 (5)	0.0196 (5)
S2	0.0595 (7)	0.0634 (7)	0.0504 (6)	−0.0189 (5)	−0.0037 (5)	0.0224 (5)
C1	0.054 (2)	0.046 (2)	0.049 (2)	0.0123 (19)	0.0080 (19)	0.0165 (18)
C2	0.060 (3)	0.039 (2)	0.047 (2)	0.0102 (19)	0.014 (2)	0.0133 (17)
C3	0.058 (3)	0.057 (3)	0.056 (2)	0.012 (2)	0.004 (2)	0.023 (2)
C4	0.054 (3)	0.060 (3)	0.082 (3)	0.013 (2)	0.011 (2)	0.024 (2)
C5	0.078 (3)	0.053 (3)	0.083 (3)	0.010 (2)	0.033 (3)	0.028 (2)
C6	0.088 (4)	0.058 (3)	0.060 (3)	0.011 (3)	0.021 (3)	0.026 (2)
C7	0.071 (3)	0.048 (2)	0.050 (2)	0.004 (2)	0.006 (2)	0.0163 (19)
C8	0.044 (2)	0.043 (2)	0.040 (2)	0.0094 (17)	0.0079 (18)	0.0112 (17)

C9	0.044 (2)	0.050 (2)	0.051 (2)	0.0031 (18)	0.0108 (18)	0.0215 (18)
C10	0.039 (2)	0.044 (2)	0.054 (2)	0.0005 (16)	0.0022 (17)	0.0224 (18)
C11	0.049 (2)	0.062 (3)	0.074 (3)	−0.001 (2)	0.013 (2)	0.032 (2)
C12	0.047 (3)	0.070 (3)	0.094 (4)	−0.012 (2)	−0.001 (2)	0.043 (3)
C13	0.078 (3)	0.048 (3)	0.081 (3)	−0.012 (2)	−0.015 (3)	0.022 (2)
C14	0.078 (3)	0.062 (3)	0.066 (3)	0.002 (3)	0.005 (3)	0.015 (2)
C15	0.052 (2)	0.053 (2)	0.063 (3)	0.000 (2)	0.007 (2)	0.020 (2)
C16	0.044 (2)	0.044 (2)	0.048 (2)	−0.0003 (17)	0.0004 (18)	0.0248 (18)

Geometric parameters (Å, º)

N1—C1	1.274 (5)	C3—H3	0.930
N1—N2	1.385 (4)	C4—C5	1.384 (6)
N2—C8	1.342 (5)	C4—H4	0.930
N2—H2	0.860	C5—C6	1.371 (7)
N3—C8	1.320 (5)	C5—H5A	0.930
N3—H3A	0.860	C6—C7	1.377 (6)
N3—H3B	0.860	C6—H6	0.930
N4—C9	1.274 (5)	C7—H7	0.930
N4—N5	1.375 (4)	C9—C10	1.453 (5)
N5—C16	1.347 (4)	C9—H9	0.930
N5—H5	0.860	C10—C11	1.385 (5)
N6—C16	1.321 (4)	C10—C15	1.390 (5)
N6—H6A	0.860	C11—C12	1.383 (6)
N6—H6B	0.860	C11—H11	0.930
S1—C8	1.693 (4)	C12—C13	1.362 (6)
S2—C16	1.674 (4)	C12—H12	0.930
C1—C2	1.466 (5)	C13—C14	1.362 (6)
C1—H1	0.930	C13—H13	0.930
C2—C3	1.375 (5)	C14—C15	1.376 (6)
C2—C7	1.388 (5)	C14—H14	0.930
C3—C4	1.377 (6)	C15—H15	0.930
C1—N1—N2	116.5 (3)	C7—C6—H6	119.8
C8—N2—N1	118.4 (3)	C6—C7—C2	120.4 (4)
C8—N2—H2	120.8	C6—C7—H7	119.8
N1—N2—H2	120.8	C2—C7—H7	119.8
C8—N3—H3A	120.0	N3—C8—N2	117.6 (3)
C8—N3—H3B	120.0	N3—C8—S1	122.5 (3)
H3A—N3—H3B	120.0	N2—C8—S1	119.9 (3)
C9—N4—N5	117.1 (3)	N4—C9—C10	120.4 (4)
C16—N5—N4	120.0 (3)	N4—C9—H9	119.8
C16—N5—H5	120.0	C10—C9—H9	119.8
N4—N5—H5	120.0	C11—C10—C15	118.8 (4)
C16—N6—H6A	120.0	C11—C10—C9	119.6 (4)
C16—N6—H6B	120.0	C15—C10—C9	121.6 (3)
H6A—N6—H6B	120.0	C12—C11—C10	120.1 (4)
N1—C1—C2	120.0 (4)	C12—C11—H11	119.9

N1—C1—H1	120.0	C10—C11—H11	119.9
C2—C1—H1	120.0	C13—C12—C11	120.1 (4)
C3—C2—C7	118.8 (4)	C13—C12—H12	119.9
C3—C2—C1	121.9 (4)	C11—C12—H12	119.9
C7—C2—C1	119.3 (4)	C14—C13—C12	120.5 (4)
C2—C3—C4	121.0 (4)	C14—C13—H13	119.8
C2—C3—H3	119.5	C12—C13—H13	119.8
C4—C3—H3	119.5	C13—C14—C15	120.4 (5)
C3—C4—C5	119.8 (5)	C13—C14—H14	119.8
C3—C4—H4	120.1	C15—C14—H14	119.8
C5—C4—H4	120.1	C14—C15—C10	120.1 (4)
C6—C5—C4	119.7 (4)	C14—C15—H15	119.9
C6—C5—H5A	120.2	C10—C15—H15	119.9
C4—C5—H5A	120.2	N6—C16—N5	116.4 (3)
C5—C6—C7	120.4 (4)	N6—C16—S2	123.6 (3)
C5—C6—H6	119.8	N5—C16—S2	120.0 (3)
C1—N1—N2—C8	−177.7 (3)	N1—N2—C8—S1	−173.6 (2)
C9—N4—N5—C16	−176.7 (3)	N5—N4—C9—C10	−175.1 (3)
N2—N1—C1—C2	−175.6 (3)	N4—C9—C10—C11	−174.2 (4)
N1—C1—C2—C3	−4.5 (6)	N4—C9—C10—C15	8.9 (6)
N1—C1—C2—C7	174.5 (4)	C15—C10—C11—C12	0.6 (6)
C7—C2—C3—C4	0.2 (6)	C9—C10—C11—C12	−176.4 (4)
C1—C2—C3—C4	179.3 (4)	C10—C11—C12—C13	0.8 (7)
C2—C3—C4—C5	0.0 (6)	C11—C12—C13—C14	−1.9 (7)
C3—C4—C5—C6	0.0 (6)	C12—C13—C14—C15	1.7 (7)
C4—C5—C6—C7	−0.2 (7)	C13—C14—C15—C10	−0.2 (7)
C5—C6—C7—C2	0.4 (6)	C11—C10—C15—C14	−0.9 (6)
C3—C2—C7—C6	−0.4 (6)	C9—C10—C15—C14	176.0 (4)
C1—C2—C7—C6	−179.5 (3)	N4—N5—C16—N6	7.6 (5)
N1—N2—C8—N3	4.7 (5)	N4—N5—C16—S2	−172.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.86	2.64	3.443 (3)	155
N3—H3*A*···S1^{ii}	0.86	2.98	3.488 (3)	120
N5—H5···S1^{ii}	0.86	2.61	3.441 (3)	162
N6—H6*B*···S2^{iii}	0.86	2.51	3.368 (3)	173

Symmetry codes: (i) −*x*+3, −*y*+2, −*z*+2; (ii) *x*−1, *y*, *z*; (iii) −*x*+2, −*y*+2, −*z*+1.