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(*E*)-1-(3-Cyanobenzylidene)thiosemicarbazide *N*,*N*-dimethylformamide solvate

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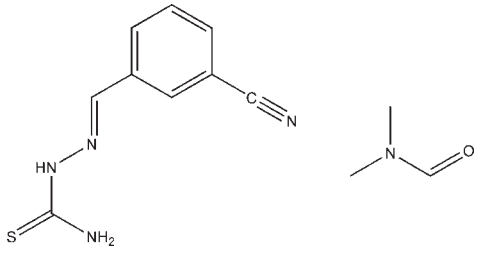
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.109; data-to-parameter ratio = 19.1.

The title compound, $C_9H_8N_4S\cdot C_3H_7NO$, adopts an *E* configuration about both the C=N and C—N bonds. Intermolecular N—H···O hydrogen bonding links the compound to the DMF solvent molecule. The crystal packing is characterized by chains of molecules linked by intermolecular N—H···S hydrogen-bonding interactions.

Related literature

For the biological activity of thiosemicarbazones, see: Lovejoy & Richardson *et al.* (2002). For a related structure, see: Wu *et al.* (2009). For comparitive geometrical parameters, see: Sutton *et al.* (1965).



Experimental

Crystal data

$C_9H_8N_4S\cdot C_3H_7NO$
M_r = 277.35
Monoclinic, $P2_1/n$
a = 7.312 (7) Å
b = 8.945 (3) Å
c = 22.316 (19) Å
β = 92.12 (2)°
V = 1458.6 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.742, T_{max} = 1.000
9561 measured reflections
3280 independent reflections
2065 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.109
S = 1.01
3280 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1	0.86	1.96	2.795 (3)	162
N4—H4A···N1^{i}	0.86	2.35	3.101 (3)	146
N4—H4B···S1^{ii}	0.86	2.59	3.364 (2)	150
C8—H8A···O1	0.93	2.54	3.293 (3)	138

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by the Natural Science Foundation (2008NXY25) of Nanjing Xiaozhuang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2253).

References

Lovejoy, D. B. & Richardson, D. R. (2002). *Blood*, **100**, 666–676.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sutton, L. E. (1965). *Tables of Interatomic Distances and Configurations in Molecules and Ions*, Suppl. London: The Chemical Society.
Wu, D.-H., Zhang, Y.-H., Li, Z.-F. & Li, Y.-H. (2009). *Acta Cryst.* E**65**, o107.

supporting information

Acta Cryst. (2010). E**66**, o421 [https://doi.org/10.1107/S160053681000214X]

(*E*)-1-(3-Cyanobenzylidene)thiosemicarbazide *N*,*N*-dimethylformamide solvate

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S1. Comment

The antiproliferative activity of a series of thiosemicarbazones has been reported (Lovejoy & Richardson, 2002). As a research on thiosemicarbazones, the synthesis and crystal structure of a new Schiff base compound derived from thiosemicarbazide and 3-cyanobenzaldehyde has been presented in this article. The crystal structure of 4-cyanobenzaldehyde thiosemicarbazone which is closely related to the title compound has been reported recently (Wu *et al.* 2009).

The thiosemicarbazone moiety in the title compound (Fig. 1) is nearly planar and shows an E configuration about both the C9—N3 and C8═N2 bonds. The C—S bond distance of 1.680 (2) Å agrees well with similar bonds in related compounds, being intermediate between 1.82 Å for a C—S single bond and 1.56 Å for a C═S double bond (Sutton *et al.* 1965). All the bond distances except for the C6—C9 (bond length, 1.448 (3) Å) fall within the normal range. The intermolecular N—H···O hydrogen bond stabilizes the molecular conformation. In the crystal packing, adjacent molecules are linked by N—H···S hydrogen bonds (Table 1 and Fig. 2) to form chains running parallel to the *a* axis. Weak interactions of the type C—H···O are also present in the structure.

S2. Experimental

The title compound was synthesized by refluxing 3-cyanobenzaldehyde (2.1 g, 16 mmol) and thiosemicarbazide (1.46 g, 16 mmol) in absolute ethanol (50 ml) for 10 h. After cooling to room temperature, the white solid formed was isolated and dried under vacuum. The title compound was isolated using column chromatography (petroleum ether: ethyl acetate-2:1). Single crystals suitable for X-ray diffraction analysis were obtained from slow evaporation of DMF solution.

S3. Refinement

H atoms were placed in calculated positions and refined using a riding model, with N—H = 0.86 Å, C—H = 0.93–0.96 Å and with U_{iso}(H) = 1.2 and 1.5 times U_{eq} of nonmethyl and methyl type H-atoms.

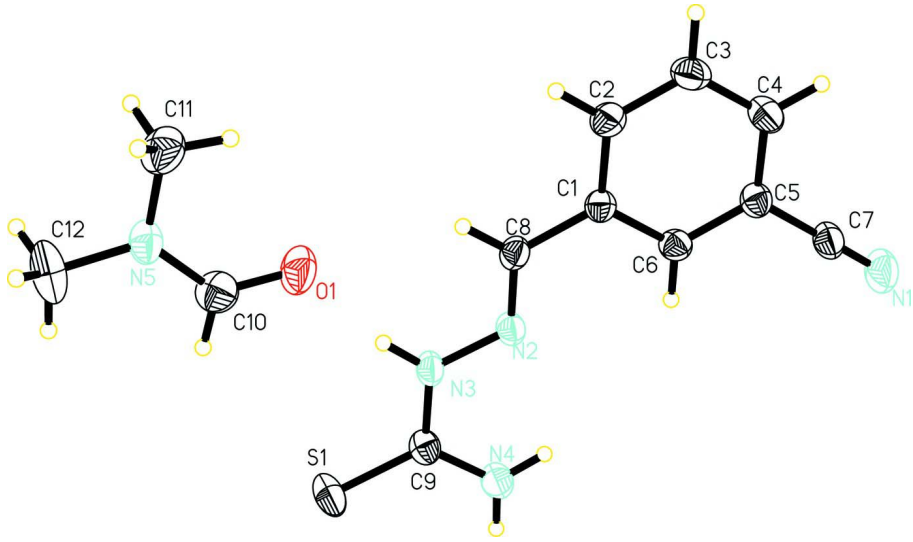

Figure 1

Perspective structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

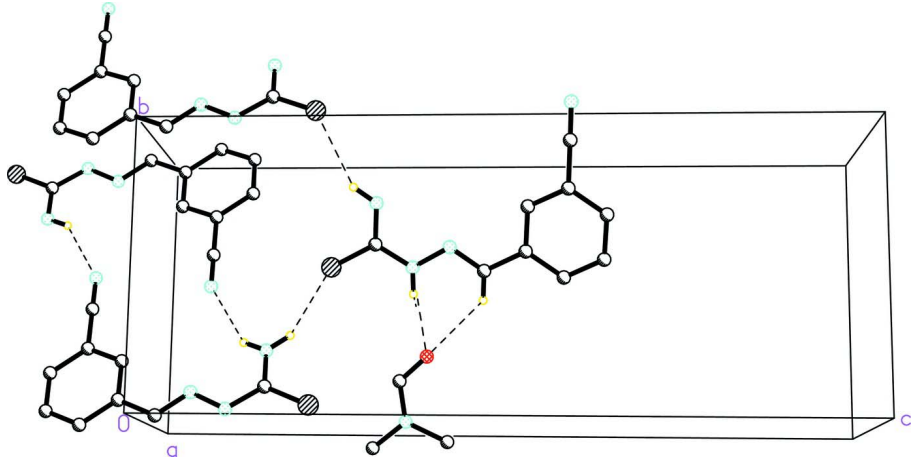

Figure 2

The crystal packing of the title compound viewed along the *a* axis showing the two-dimensionnal hydrogen bondings network. H-atoms non involved in H-bonding interactions have been excluded for clarity.

(*E*)-1-(3-Cyanobenzylidene)thiosemicarbazide *N*,*N*-dimethylformamide solvate

Crystal data

$C_9H_8N_4S·C_3H_7NO$
$M_r = 277.35$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.312$ (7) Å
$b = 8.945$ (3) Å
$c = 22.316$ (19) Å
$\beta = 92.12$ (2)°
$V = 1458.6$ (19) Å^3
$Z = 4$

$F(000) = 584$
$D_x = 1.263$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2851 reflections
$\theta = 2.3$–27.4°
$\mu = 0.22$ mm^{-1}
$T = 293$ K
Block, pale yellow
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.742, T_{max} = 1.000
9561 measured reflections
3280 independent reflections
2065 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.4°, θ_{min} = 2.5°
$h = -7 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -28 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.109
S = 1.01
3280 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.020P)^2 + 0.850P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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}
S1	0.60691 (11)	0.46135 (8)	0.74146 (3)	0.0636 (2)
N2	0.7389 (2)	0.40996 (19)	0.57477 (8)	0.0425 (4)
N3	0.6727 (2)	0.4707 (2)	0.62652 (8)	0.0451 (5)
H3A	0.6198	0.5566	0.6258	0.054*
C6	0.8765 (3)	0.2925 (2)	0.46844 (10)	0.0430 (5)
H6A	0.8857	0.2327	0.5025	0.052*
N4	0.7782 (3)	0.2634 (2)	0.67556 (9)	0.0585 (6)
H4A	0.8181	0.2320	0.6420	0.070*
H4B	0.7939	0.2102	0.7074	0.070*
C8	0.7256 (3)	0.4903 (2)	0.52748 (10)	0.0428 (5)
H8A	0.6724	0.5846	0.5288	0.051*
C9	0.6924 (3)	0.3936 (2)	0.67810 (10)	0.0441 (5)
C7	1.0229 (3)	0.0938 (3)	0.41270 (10)	0.0518 (6)
C1	0.7947 (3)	0.4329 (2)	0.47102 (9)	0.0402 (5)
N1	1.0774 (3)	−0.0249 (3)	0.41178 (10)	0.0714 (7)
C5	0.9444 (3)	0.2423 (2)	0.41459 (10)	0.0444 (5)

C2	0.7832 (3)	0.5198 (3)	0.41938 (10)	0.0501 (6)
H2B	0.7295	0.6140	0.4207	0.060*
C3	0.8504 (3)	0.4685 (3)	0.36610 (10)	0.0565 (6)
H3B	0.8407	0.5281	0.3320	0.068*
C4	0.9316 (3)	0.3298 (3)	0.36311 (10)	0.0535 (6)
H4C	0.9770	0.2953	0.3273	0.064*
N5	0.4289 (3)	0.9814 (2)	0.63750 (9)	0.0565 (5)
C10	0.4945 (4)	0.8452 (3)	0.64567 (13)	0.0641 (7)
H10A	0.4904	0.8055	0.6841	0.077*
O1	0.5606 (3)	0.76602 (19)	0.60695 (9)	0.0718 (6)
C11	0.4348 (4)	1.0502 (3)	0.57878 (13)	0.0756 (8)
H11A	0.4869	0.9813	0.5512	0.113*
H11B	0.5085	1.1389	0.5813	0.113*
H11C	0.3129	1.0759	0.5650	0.113*
C12	0.3501 (4)	1.0680 (4)	0.68517 (15)	0.0925 (11)
H12A	0.3521	1.0099	0.7214	0.139*
H12B	0.2261	1.0936	0.6740	0.139*
H12C	0.4202	1.1577	0.6917	0.139*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0917 (5)	0.0516 (4)	0.0489 (4)	−0.0029 (4)	0.0207 (3)	−0.0114 (3)
N2	0.0512 (12)	0.0372 (9)	0.0396 (10)	0.0037 (8)	0.0069 (9)	−0.0025 (8)
N3	0.0550 (12)	0.0366 (9)	0.0444 (11)	0.0082 (9)	0.0102 (9)	−0.0053 (8)
C6	0.0502 (14)	0.0383 (12)	0.0403 (12)	0.0002 (10)	0.0011 (10)	0.0022 (9)
N4	0.0848 (16)	0.0461 (11)	0.0454 (12)	0.0152 (11)	0.0121 (11)	0.0056 (9)
C8	0.0450 (13)	0.0359 (12)	0.0477 (14)	0.0064 (10)	0.0030 (10)	−0.0018 (10)
C9	0.0508 (15)	0.0351 (11)	0.0465 (13)	−0.0047 (10)	0.0054 (11)	−0.0038 (10)
C7	0.0604 (16)	0.0490 (14)	0.0468 (14)	0.0050 (12)	0.0114 (12)	−0.0048 (11)
C1	0.0421 (13)	0.0373 (12)	0.0413 (12)	0.0011 (9)	0.0014 (10)	−0.0015 (9)
N1	0.0898 (18)	0.0539 (14)	0.0718 (16)	0.0190 (13)	0.0214 (13)	−0.0032 (12)
C5	0.0486 (14)	0.0392 (12)	0.0454 (13)	0.0005 (10)	0.0031 (11)	−0.0050 (10)
C2	0.0603 (16)	0.0407 (12)	0.0492 (14)	0.0063 (11)	−0.0003 (12)	0.0025 (11)
C3	0.0730 (18)	0.0554 (15)	0.0408 (14)	0.0039 (13)	0.0006 (12)	0.0081 (12)
C4	0.0642 (17)	0.0558 (15)	0.0411 (14)	−0.0010 (13)	0.0075 (12)	−0.0042 (11)
N5	0.0650 (14)	0.0431 (11)	0.0621 (14)	0.0022 (10)	0.0117 (11)	−0.0079 (10)
C10	0.074 (2)	0.0515 (16)	0.0665 (18)	−0.0059 (14)	0.0011 (15)	0.0052 (13)
O1	0.0870 (15)	0.0432 (10)	0.0860 (14)	0.0116 (10)	0.0131 (11)	−0.0066 (10)
C11	0.088 (2)	0.0541 (17)	0.086 (2)	0.0066 (15)	0.0132 (17)	0.0126 (15)
C12	0.095 (2)	0.083 (2)	0.102 (3)	−0.0076 (19)	0.034 (2)	−0.0443 (19)

Geometric parameters (Å, º)

S1—C9	1.680 (2)	C2—C3	1.382 (3)
N2—C8	1.277 (3)	C2—H2B	0.9300
N2—N3	1.380 (2)	C3—C4	1.378 (3)
N3—C9	1.345 (3)	C3—H3B	0.9300

N3—H3A	0.8600	C4—H4C	0.9300
C6—C5	1.392 (3)	N5—C10	1.320 (3)
C6—C1	1.393 (3)	N5—C11	1.450 (3)
C6—H6A	0.9300	N5—C12	1.452 (3)
N4—C9	1.325 (3)	C10—O1	1.230 (3)
N4—H4A	0.8600	C10—H10A	0.9300
N4—H4B	0.8600	C11—H11A	0.9600
C8—C1	1.467 (3)	C11—H11B	0.9600
C8—H8A	0.9300	C11—H11C	0.9600
C7—N1	1.135 (3)	C12—H12A	0.9600
C7—C5	1.447 (3)	C12—H12B	0.9600
C1—C2	1.390 (3)	C12—H12C	0.9600
C5—C4	1.390 (3)		

C8—N2—N3	116.85 (18)	C1—C2—H2B	119.5
C9—N3—N2	118.97 (18)	C4—C3—C2	120.5 (2)
C9—N3—H3A	120.5	C4—C3—H3B	119.7
N2—N3—H3A	120.5	C2—C3—H3B	119.7
C5—C6—C1	119.6 (2)	C3—C4—C5	118.9 (2)
C5—C6—H6A	120.2	C3—C4—H4C	120.6
C1—C6—H6A	120.2	C5—C4—H4C	120.6
C9—N4—H4A	120.0	C10—N5—C11	119.6 (2)
C9—N4—H4B	120.0	C10—N5—C12	122.9 (3)
H4A—N4—H4B	120.0	C11—N5—C12	117.5 (2)
N2—C8—C1	119.7 (2)	O1—C10—N5	125.8 (3)
N2—C8—H8A	120.2	O1—C10—H10A	117.1
C1—C8—H8A	120.2	N5—C10—H10A	117.1
N4—C9—N3	116.8 (2)	N5—C11—H11A	109.5
N4—C9—S1	123.00 (18)	N5—C11—H11B	109.5
N3—C9—S1	120.24 (17)	H11A—C11—H11B	109.5
N1—C7—C5	177.1 (3)	N5—C11—H11C	109.5
C2—C1—C6	118.9 (2)	H11A—C11—H11C	109.5
C2—C1—C8	120.3 (2)	H11B—C11—H11C	109.5
C6—C1—C8	120.8 (2)	N5—C12—H12A	109.5
C4—C5—C6	121.1 (2)	N5—C12—H12B	109.5
C4—C5—C7	120.5 (2)	H12A—C12—H12B	109.5
C6—C5—C7	118.4 (2)	N5—C12—H12C	109.5
C3—C2—C1	121.0 (2)	H12A—C12—H12C	109.5
C3—C2—H2B	119.5	H12B—C12—H12C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1	0.86	1.96	2.795 (3)	162
N4—H4*A*···N2	0.86	2.25	2.610 (3)	105
N4—H4*A*···N1^{i}	0.86	2.35	3.101 (3)	146
N4—H4*B*···S1^{ii}	0.86	2.59	3.364 (2)	150

C8—H8*A*···O1	0.93	2.54	3.293 (3)	138
C11—H11*A*···O1	0.96	2.34	2.767 (3)	106

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