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N,*N*-Dimethyl-2-[(1*E*)-({[(methylsulfanyl)methanethioyl]amino}imino)methyl]aniline

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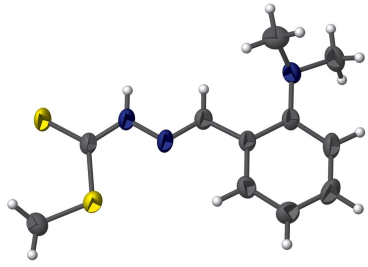
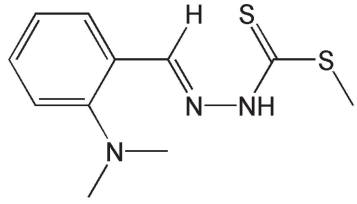
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_{15}N_3S_2$, the dithiocarbazate moiety is rotated by 18.73 (8)° with respect to the benzene ring. The dithiocarbazate group adopts an *E* configuration with respect to the C=N bond of the benzylidene group. Furthermore, in the solid state the compound exists in the thione tautomeric form. In the crystal, molecules are linked by pairs of weak N—H···S hydrogen bonds, forming inversion dimers which are arranged in layers parallel to (010).

3D view



Chemical scheme



Structure description

Dithiocarbazate derivatives remain of interest to researchers because of their extensive variations in structure and promising biological and catalytic activities (Low *et al.*, 2014). Metal complexes of ligands derived from dithiocarbazic acids have created a significant interest in their coordination chemistry (Mahapatra *et al.*, 2013). As a part of our ongoing research on such molecules, we report herein on the synthesis and crystal structure of the title compound.

The molecule is not completely planar (r.m.s. deviation for all non-H atoms 0.375 Å). The thione sulfur atom (S1) is positioned *trans* to the azomethine nitrogen (N1) atom, Fig. 1. The C═S and C—S bond lengths of 1.6606 (17) and 1.7462 (19) Å, respectively, are of the order of those in related dithiocarbazate based Schiff bases (Basha *et al.*, 2012). The observed bond lengths are intermediate between C—S and C═S bonds, indicating conjugation effects along the ═N—NH—C(═S)—SCH_3 chain.

In the crystal, the molecules are linked by N—H···S hydrogen bonds (Table 1, Fig. 2), forming inversion dimers.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H9···S1^{i}	0.86	2.62	3.4566 (16)	166

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

Synthesis and crystallization

The synthesis of the title compound is illustrated in Fig. 3. A solution of 2-dimethylamino benzaldehyde (0.298 g, 2 mmol) in methanol (10 ml) was added to a stirred solution of *S*-methyl dithiocarbazate (0.25 g, 2 mmol) in ethanol (15 ml). The mixture was stirred for 10 min then refluxed for 6 h. The reaction mixture was then cooled to room temperature and the yellow solid obtained was filtered off, washed with cold methanol and dried under vacuum over anhydrous $CaCl_2$. This solid was recrystallized from methanol, yielding needle-shaped crystals that were suitable for X-ray diffraction studies.

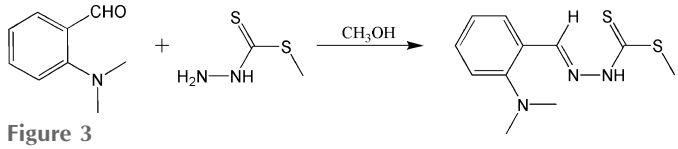


Figure 3
Synthesis of the title compound.

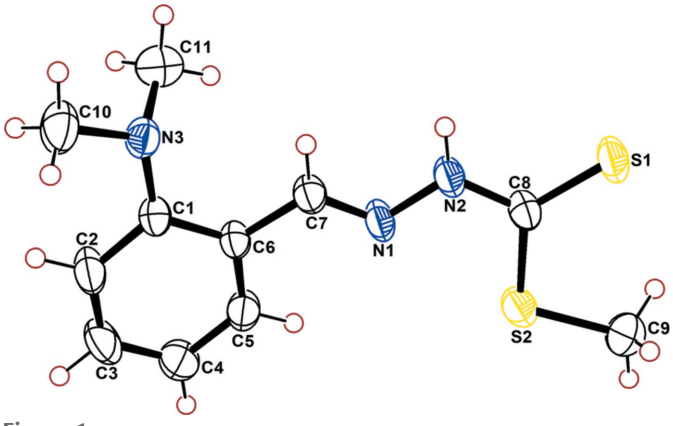


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

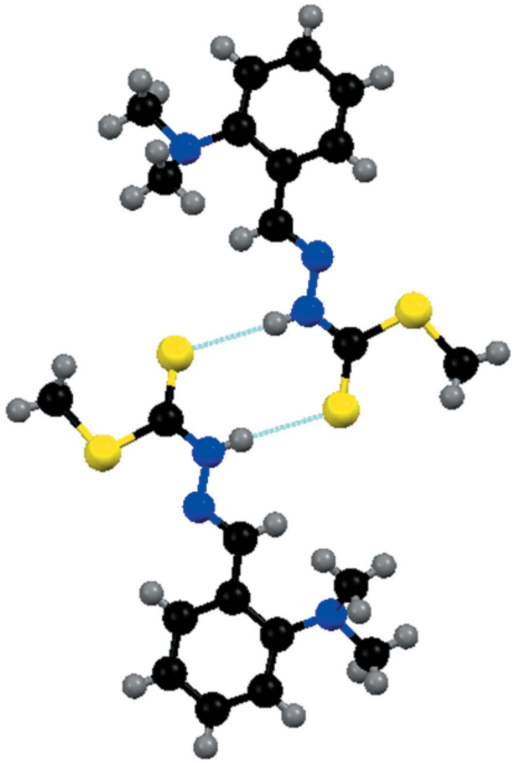


Figure 2
Dimer formation through N—H···S interactions.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{15}N_3S_2$
M_r	253.38
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	20.5182 (12), 7.6402 (6), 16.6523 (12)
β (°)	96.863 (6)
V (Å^3)	2591.8 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.39
Crystal size (mm)	0.46 × 0.40 × 0.38
Data collection	
Diffractometer	Agilent EOS, Gemini
Absorption correction	Multi-scan (SCALE3 ABSPACK; Agilent, 2015)
T_{min}, T_{max}	0.513, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4778, 2638, 2136
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.097, 1.04
No. of reflections	2638
No. of parameters	148
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.24

Computer programs: *CrysAlis PRO* (Agilent, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *ORTEP-3 for Windows* (Farrugia, 2012).

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full crystallographic data

IUCrData (2018). **3**, x180461 [https://doi.org/10.1107/S2414314618004613]

N,*N*-Dimethyl-2-[(1*E*)-({[(methylsulfanyl)methanethioyl]amino}imino)methyl]-aniline

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_N_,_N_-Dimethyl-2-[(1_E_)-({[(methylsulfanyl)methanethioyl]amino}imino)methyl]aniline

Crystal data

$C_{11}H_{15}N_3S_2$
M_r = 253.38
Monoclinic, $C2/c$
a = 20.5182 (12) Å
b = 7.6402 (6) Å
c = 16.6523 (12) Å
β = 96.863 (6)°
V = 2591.8 (3) Å^3
Z = 8
$F(000)$ = 1072
D_x = 1.299 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2638 reflections
θ = 3.8–26.4°
μ = 0.39 mm^{-1}
T = 293 K
Needle, yellow
0.46 × 0.40 × 0.38 mm

Data collection

Agilent EOS, Gemini diffractometer
profile data from $\theta/2\theta$ scans
Absorption correction: multi-scan (SCALE3 ABSPACK; Agilent, 2015)
T_{min} = 0.513, T_{max} = 0.747
4778 measured reflections
2638 independent reflections
2136 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.4°, θ_{min} = 3.8°
h = −25→24
k = −9→9
l = −20→11
4386 standard reflections every 10 reflections
intensity decay: 5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.04
2638 reflections
148 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 1.4528P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S2	0.41502 (2)	0.68062 (8)	0.46488 (3)	0.04706 (17)
S1	0.53348 (2)	0.74013 (8)	0.37613 (3)	0.05367 (18)
N3	0.22176 (7)	0.6136 (2)	0.10219 (9)	0.0389 (4)
N2	0.41551 (7)	0.6548 (2)	0.31042 (9)	0.0418 (4)
H9	0.4306	0.6560	0.2644	0.050*
N1	0.35047 (6)	0.6156 (2)	0.31475 (9)	0.0408 (4)
C6	0.24224 (8)	0.5859 (2)	0.24910 (10)	0.0332 (4)
C1	0.19790 (8)	0.5987 (2)	0.17786 (10)	0.0343 (4)
C7	0.31255 (8)	0.6169 (2)	0.24855 (11)	0.0367 (4)
H7	0.3294	0.6374	0.2000	0.044*
C8	0.45484 (8)	0.6910 (2)	0.37801 (11)	0.0367 (4)
C5	0.21800 (9)	0.5579 (3)	0.32248 (11)	0.0421 (4)
H6	0.2473	0.5421	0.3690	0.051*
C4	0.15177 (9)	0.5530 (3)	0.32794 (13)	0.0508 (5)
H5	0.1363	0.5341	0.3775	0.061*
C2	0.13109 (9)	0.5980 (3)	0.18530 (12)	0.0485 (5)
H3	0.1010	0.6125	0.1394	0.058*
C10	0.17401 (10)	0.6747 (3)	0.03593 (12)	0.0568 (6)
H16A	0.1403	0.5881	0.0244	0.085*
H16B	0.1956	0.6937	−0.0113	0.085*
H16C	0.1547	0.7823	0.0512	0.085*
C11	0.25651 (11)	0.4594 (3)	0.07815 (13)	0.0552 (5)
H15A	0.2866	0.4196	0.1230	0.083*
H15B	0.2803	0.4888	0.0338	0.083*
H15C	0.2255	0.3684	0.0619	0.083*
C9	0.47913 (10)	0.7383 (3)	0.54339 (12)	0.0541 (5)
H12A	0.5182	0.6740	0.5362	0.081*
H12B	0.4656	0.7103	0.5951	0.081*
H12C	0.4879	0.8615	0.5409	0.081*
C3	0.10867 (9)	0.5764 (3)	0.25900 (13)	0.0554 (6)
H4	0.0638	0.5776	0.2624	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S2	0.0306 (2)	0.0678 (4)	0.0429 (3)	−0.0013 (2)	0.00473 (19)	0.0017 (2)
S1	0.0264 (2)	0.0880 (4)	0.0456 (3)	−0.0157 (2)	0.0003 (2)	0.0005 (3)
N3	0.0346 (8)	0.0473 (9)	0.0336 (8)	−0.0023 (7)	−0.0012 (6)	0.0035 (7)
N2	0.0237 (7)	0.0627 (10)	0.0384 (8)	−0.0056 (7)	0.0010 (6)	0.0039 (8)
N1	0.0224 (7)	0.0569 (10)	0.0420 (8)	−0.0057 (7)	−0.0008 (6)	0.0041 (7)
C6	0.0259 (8)	0.0370 (9)	0.0357 (9)	−0.0046 (7)	0.0003 (7)	0.0011 (7)
C1	0.0289 (8)	0.0352 (9)	0.0376 (9)	−0.0047 (7)	−0.0006 (7)	0.0018 (8)
C7	0.0289 (8)	0.0438 (10)	0.0368 (9)	−0.0036 (8)	0.0019 (7)	0.0039 (8)
C8	0.0267 (8)	0.0428 (10)	0.0397 (9)	−0.0005 (7)	0.0002 (7)	0.0054 (8)
C5	0.0351 (9)	0.0540 (11)	0.0359 (10)	−0.0072 (8)	−0.0011 (7)	0.0005 (8)

C4	0.0403 (10)	0.0688 (14)	0.0452 (11)	−0.0109 (10)	0.0126 (8)	0.0006 (10)
C2	0.0277 (9)	0.0663 (13)	0.0491 (11)	−0.0051 (9)	−0.0051 (8)	0.0071 (10)
C10	0.0465 (11)	0.0782 (15)	0.0428 (11)	−0.0069 (11)	−0.0069 (9)	0.0143 (11)
C11	0.0596 (13)	0.0564 (13)	0.0513 (12)	0.0007 (11)	0.0132 (10)	−0.0033 (10)
C9	0.0476 (12)	0.0704 (14)	0.0424 (11)	0.0024 (10)	−0.0018 (9)	−0.0041 (10)
C3	0.0257 (9)	0.0790 (16)	0.0625 (13)	−0.0057 (10)	0.0089 (9)	0.0060 (12)

Geometric parameters (Å, º)

S2—C8	1.7462 (19)	C5—H6	0.9300
S2—C9	1.795 (2)	C4—C3	1.375 (3)
S1—C8	1.6606 (17)	C4—H5	0.9300
N3—C1	1.410 (2)	C2—C3	1.371 (3)
N3—C11	1.458 (3)	C2—H3	0.9300
N3—C10	1.462 (2)	C10—H16A	0.9600
N2—C8	1.333 (2)	C10—H16B	0.9600
N2—N1	1.3781 (19)	C10—H16C	0.9600
N2—H9	0.8600	C11—H15A	0.9600
N1—C7	1.271 (2)	C11—H15B	0.9600
C6—C5	1.390 (2)	C11—H15C	0.9600
C6—C1	1.410 (2)	C9—H12A	0.9600
C6—C7	1.463 (2)	C9—H12B	0.9600
C1—C2	1.391 (2)	C9—H12C	0.9600
C7—H7	0.9300	C3—H4	0.9300
C5—C4	1.373 (2)		
C8—S2—C9	102.56 (9)	C3—C4—H5	120.5
C1—N3—C11	114.42 (15)	C3—C2—C1	121.32 (17)
C1—N3—C10	115.30 (15)	C3—C2—H3	119.3
C11—N3—C10	110.87 (16)	C1—C2—H3	119.3
C8—N2—N1	119.56 (15)	N3—C10—H16A	109.5
C8—N2—H9	120.2	N3—C10—H16B	109.5
N1—N2—H9	120.2	H16A—C10—H16B	109.5
C7—N1—N2	116.69 (15)	N3—C10—H16C	109.5
C5—C6—C1	119.20 (15)	H16A—C10—H16C	109.5
C5—C6—C7	119.16 (15)	H16B—C10—H16C	109.5
C1—C6—C7	121.42 (15)	N3—C11—H15A	109.5
C2—C1—C6	117.94 (16)	N3—C11—H15B	109.5
C2—C1—N3	122.09 (15)	H15A—C11—H15B	109.5
C6—C1—N3	119.97 (15)	N3—C11—H15C	109.5
N1—C7—C6	119.68 (16)	H15A—C11—H15C	109.5
N1—C7—H7	120.2	H15B—C11—H15C	109.5
C6—C7—H7	120.2	S2—C9—H12A	109.5
N2—C8—S1	121.50 (14)	S2—C9—H12B	109.5
N2—C8—S2	113.21 (12)	H12A—C9—H12B	109.5
S1—C8—S2	125.29 (11)	S2—C9—H12C	109.5
C4—C5—C6	121.56 (17)	H12A—C9—H12C	109.5
C4—C5—H6	119.2	H12B—C9—H12C	109.5

C6—C5—H6	119.2	C2—C3—C4	120.80 (17)
C5—C4—C3	118.95 (18)	C2—C3—H4	119.6
C5—C4—H5	120.5	C4—C3—H4	119.6
C8—N2—N1—C7	166.28 (17)	N1—N2—C8—S1	−179.69 (13)
C5—C6—C1—C2	5.6 (3)	N1—N2—C8—S2	0.3 (2)
C7—C6—C1—C2	−168.92 (17)	C9—S2—C8—N2	−179.01 (15)
C5—C6—C1—N3	−175.15 (16)	C9—S2—C8—S1	1.01 (16)
C7—C6—C1—N3	10.3 (3)	C1—C6—C5—C4	−4.0 (3)
C11—N3—C1—C2	−114.6 (2)	C7—C6—C5—C4	170.62 (19)
C10—N3—C1—C2	15.7 (3)	C6—C5—C4—C3	0.0 (3)
C11—N3—C1—C6	66.2 (2)	C6—C1—C2—C3	−3.4 (3)
C10—N3—C1—C6	−163.50 (17)	N3—C1—C2—C3	177.38 (19)
N2—N1—C7—C6	−176.64 (15)	C1—C2—C3—C4	−0.6 (3)
C5—C6—C7—N1	0.9 (3)	C5—C4—C3—C2	2.4 (3)
C1—C6—C7—N1	175.40 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H9···S1^{i}	0.86	2.62	3.4566 (16)	166

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.