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Methyl β-*N*-(3-nitrophenylmethylene)-dithiocarbazate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.079
Data-to-parameter ratio = 17.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Crystals of the title compound, $C_9H_9N_3O_2S_2$, were obtained from a condensation reaction of *S*-methyl dithiocarbazate and 3-nitrobenzaldehyde. The planar dithiocarbazate moiety subtends an angle of 10.54 (8)° with respect to the plane of the nitrophenyl ring. Electron delocalization occurs between the imino and dithiocarboxyl groups. The partially overlapped arrangement of parallel benzene rings of neighboring molecules, with a face-to-face distance of 3.343 (8) Å, suggests the existence of π–π stacking.

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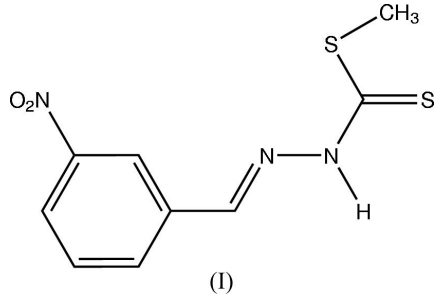
Comment

Phenylhydrazone and its derivatives have attracted our attention as they show potential applications in the biological field (Okabe *et al.*, 1993; Hu *et al.*, 2001). As part of our ongoing investigation into the anticancer properties of phenylhydrazone, the title compound, (I), has been prepared and its structure is presented here.



(I)

The molecular structure of (I) is shown in Fig. 1. The dithiocarbazate moiety is planar; its mean plane subtends an

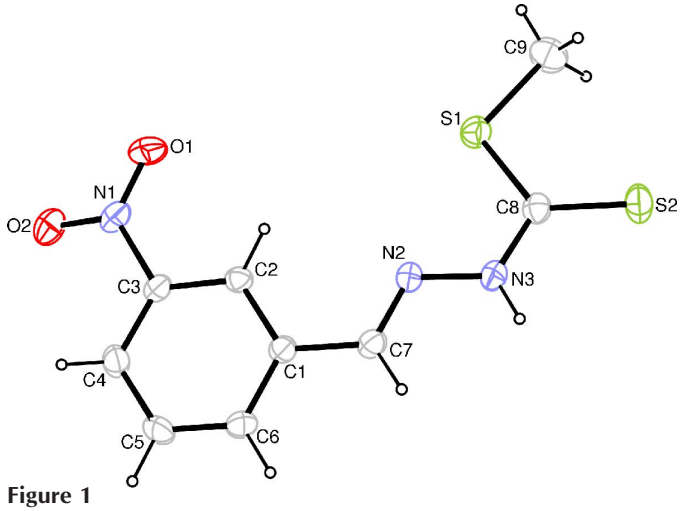


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids.

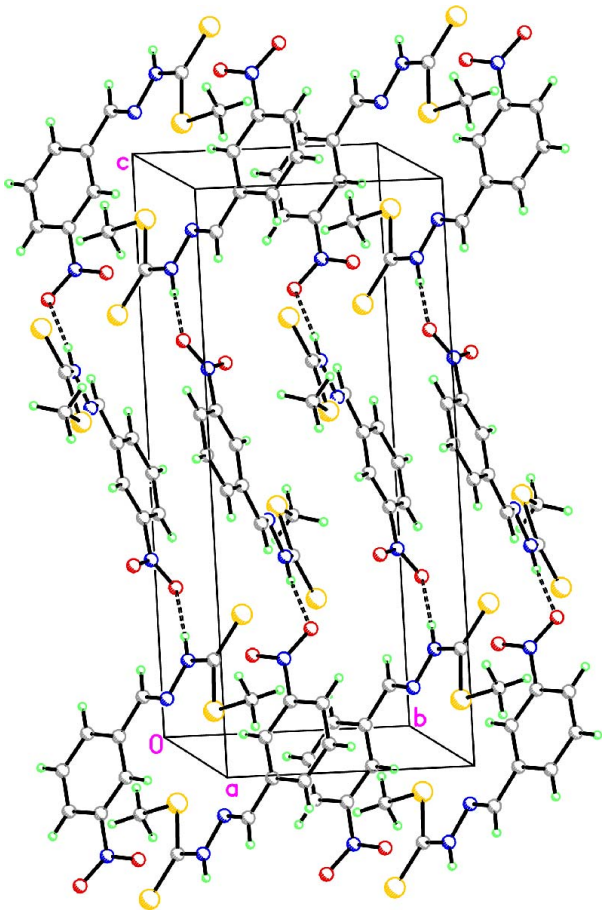


Figure 2
The crystal packing, showing the hydrogen bonding (dashed lines) and parallel arrangement of benzene rings of neighboring molecules.

angle of 10.54 (8)° with respect to the plane of the benzene ring. This differs from the situation in the related 4-nitrophenyl isomer, in which both sections of the molecule are coplanar (Duan *et al.*, 1997). The relatively short C8—N3 bond distance (Table 1) suggests a degree of electron delocalization between the imino and dithiocarboxyl groups. The nitro group is tilted out of the benzene plane, with a dihedral angle of 10.6 (2)°, which may be due to hydrogen bonding between atom O2 of the nitro substituent and the imino group of a neighboring molecule (Table 2 and Fig. 2).

A partially overlapped arrangement of parallel benzene rings [symmetry code: (ii) $1-x, -y, 1-z$] is observed in the crystal structur (Fig. 3). The face-to-face distance of 3.343 (8) Å clearly suggests the existence ofe π–π stacking between benzene rings.

Experimental

Methyl dithiocarbazate was synthesized in the manner reported previously (Hu *et al.*, 2001). Methyl dithiocarbazate (1.24 g, 10 mmol) and 3-nitrobenzaldehyde (1.50 g, 10 mmol) were dissolved in ethanol (10 ml) and refluxed for 4 h. Fine yellow crystals appeared on cooling. They were separated and washed with cold water three times. Single crystals of (I) were obtained by recrystallization from absolute ethanol.

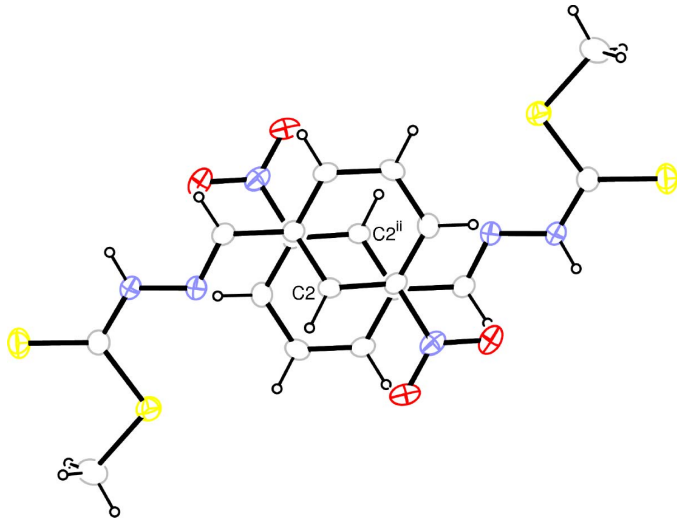


Figure 3
π–π stacking between benzene rings [symmetry code: (i) $1-x, -y, 1-z$].

Crystal data

$C_9H_9N_3O_2S_2$
$M_r = 255.31$
Monoclinic, $P2_1/c$
$a = 8.7176$ (4) Å
$b = 7.5589$ (3) Å
$c = 17.5879$ (7) Å
$\beta = 100.666$ (2)°
$V = 1138.94$ (8) Å^3
$Z = 4$
$D_x = 1.489$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4542 reflections
$\theta = 2.4$–27.0°
$\mu = 0.46$ mm^{-1}
$T = 295$ (2) K
Needle, yellow
0.50 × 0.13 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.770$, $T_{max} = 0.952$
4646 measured reflections
2611 independent reflections
1703 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.079$
$S = 0.88$
2611 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.7457 (17)	N2—N3	1.3707 (18)
S1—C9	1.791 (2)	N3—C8	1.340 (2)
S2—C8	1.6559 (17)	C1—C7	1.462 (2)
N2—C7	1.270 (2)		
C8—S1—C9	102.10 (9)	S1—C8—S2	125.87 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2^{i}	0.86	2.29	3.125 (2)	163

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

Methyl H atoms were placed in calculated positions (C—H = 0.96 Å) and torsion angle was refined to fit the electron density, with $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in calculated positions (C—H = 0.93 Å and N—H = 0.86 Å) and included in the final cycles of refinement as riding, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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