

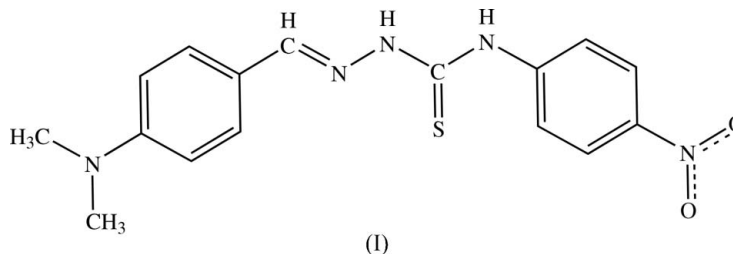
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Key indicators

Single-crystal X-ray study
T = 123 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.049
wR factor = 0.141
Data-to-parameter ratio = 13.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-[4-(Dimethylamino)benzylidene]-4-(4-nitro-
phenyl)thiosemicarbazideIn the title compound, $\text{C}_{16}\text{H}_{17}\text{N}_5\text{O}_2\text{S}$, the configuration is *trans*
about the formal $\text{C}=\text{N}$ bond. The molecular conformation is
stabilized by an intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond.Received 28 November 2006
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Comment

Thiosemicarbazones are useful chelating ligands for transition
metal ions and possess many biological activities (Chikate *et al.*,
2005). However, there are fewer reports of thio-
semicarbazones acting as anion acceptors (Krisitin, 2005). As
part of our work in this area, we report here the synthesis and
structure of the title compound, (I) (Fig. 1). The introduction
of a strong electron-donating group such as the *N,N*-
dimethylanilino group through a double bond imparts new
properties that are not commonly observed for the parent
thiourea.The configuration is *trans* about the $\text{C9}=\text{N2}$ double bond.
The $\text{C}-\text{N}$ and $\text{N}-\text{N}$ bond lengths in the linking unit (Table 1)
imply significant electron delocalization and the $\text{C9}/\text{N2}/\text{N3}/$
 $\text{C10}/\text{N4}$ fragment is close to planar (r.m.s. deviation =
 0.097 \AA). The bond-angle sum at N1 of 360.0° implies sp^2
hybridization for this atom. The dihedral angle between the
 $\text{C3}-\text{C8}$ and $\text{C11}-\text{C16}$ benzene rings is $19.10 (11)^\circ$.An intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond (Table 1)
helps to stabilize the molecular conformation. An inter-
molecular $\text{N}-\text{H}\cdots\text{S}$ interaction is also present.

Experimental

4-Isothiocyanatonitrobenzene (0.720 g, 4 mmol) was dissolved in
ethanol (30 ml), and the resulting solution was then slowly added to
50% hydrazine hydrate (1.00 g, 10 mmol) in ethanol (10 ml). The
mixed solution was stirred at room temperature for 2 h, and the
resulting yellow solid [4-(4-nitrophenyl)thiosemicarbazide] was
collected by filtration and recrystallized from ethanol. 4-(4-Nitro-
phenyl)thiosemicarbazide (0.424 g, 2 mmol) was then dissolved in
ethanol (20 ml) and added to a refluxing ethanol solution of *p*-
dimethylaminobenzaldehyde (0.298 g, 2 mmol). The mixed solution
was refluxed for a further 6 h, forming a red precipitate which was
then filtered off under reduced pressure and washed several times

with ethanol. The product was recrystallized from ethanol, and dried under vacuum for 4 h. Red block-shaped crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution at room temperature. Elemental analysis calculated for $C_{16}H_{17}N_5O_2S$ (%): C 55.96, H 4.96, N 20.39, S 9.34; found: C 55.74, H 4.95, N 20.43, S 10.31.

Crystal data

$C_{16}H_{17}N_5O_2S$
 $M_r = 343.41$
 Triclinic, $P\bar{1}$
 $a = 7.213$ (2) Å
 $b = 7.487$ (2) Å
 $c = 15.452$ (4) Å
 $\alpha = 80.920$ (4)°
 $\beta = 80.634$ (4)°
 $\gamma = 89.247$ (4)°

$V = 812.9$ (3) Å³
 $Z = 2$
 $D_x = 1.403$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹
 $T = 123$ (2) K
 Block, red
 $0.39 \times 0.26 \times 0.19$ mm

Data collection

Bruker SMART APEX 2000 CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.920$, $T_{\max} = 0.966$

5827 measured reflections
 2820 independent reflections
 2567 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.141$
 $S = 1.05$
 2820 reflections
 217 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2 + 0.7024P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.54$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.55$ e Å⁻³

Table 1

Selected bond lengths (Å).

N2—C9	1.278 (3)	N4—C10	1.351 (3)
N2—N3	1.369 (3)	N4—C11	1.393 (3)
N3—C10	1.344 (3)	C6—C9	1.443 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3A \cdots S1 ⁱ	0.88	2.44	3.315 (2)	170
N4—H4A \cdots N2	0.88	2.07	2.543 (3)	113

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

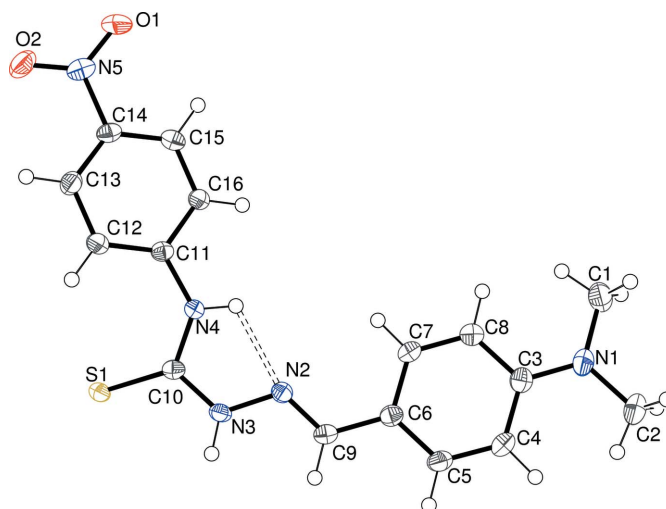


Figure 1

View of the molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The intramolecular hydrogen bond is indicated by dashed lines.

All H atoms were positioned geometrically ($C-H = 0.95-0.98$ Å and $N-H = 0.88$ Å) and refined as riding, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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