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Key indicators

Single-crystal X-ray study

T = 120 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.046

wR factor = 0.132

Data-to-parameter ratio = 17.4

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N'*-(3,5-Dinitrobenzoyl)-*N,N*-diethylthiourea

The molecular structure of the title compound, $\text{C}_{12}\text{H}_{14}\text{N}_4\text{O}_5\text{S}$, shows a twisted conformation of the carbonyl and thiocarbonyl groups. In the crystal packing, molecules are linked *via* intermolecular hydrogen bonds into columns extending in the [100] and [001] directions.

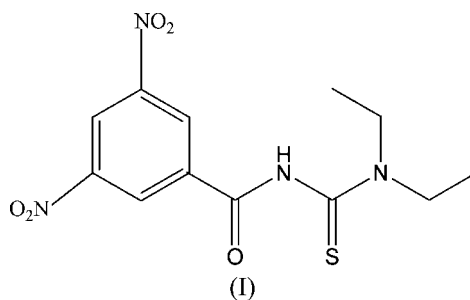
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Comment

Thiourea derivatives are versatile ligands, able to coordinate to metal centres. Recently, we discussed a novel series of thiourea derivatives and their metal complexes (Emen *et al.*, 2005). In this context, one of these new derivatives is the title compound, reported here. Its molecular structure (Fig. 1) is closely related to that of the 3,5-di(trifluoromethyl)-substituted compound (Kampf *et al.*, 2004). Bond lengths and angles of the thiourea group agree well with those expected from other thiourea compounds (Arslan *et al.*, 2006) and need not be discussed further. The conformation of the molecule with respect to the carbonyl and thiocarbonyl part is twisted, as reflected by the C8–N3–C7–O5 and C7–N3–C8–N4 torsion angles of $17.6(3)^\circ$ and $-139.7(2)^\circ$, respectively. The C2–C1–C7–O5 torsion angle set up by the aromatic ring and the carbonyl group is $22.6(2)^\circ$. Both NO_2 groups are coplanar with the C1–C6 ring; the largest deviation from this plane is $0.181(2) \text{ \AA}$ for atom O3.

The crystal packing (Fig. 2) is determined by strong intermolecular hydrogen bonds (Table 1), which link molecules into infinite columns extending along [100] and [001]. In the [010] direction these columns are separated by head-to-head orientation of the nitro groups.



Experimental

The title compound was prepared by converting 3,5-dinitrobenzoyl chloride into 3,5-dinitrobenzoyl isothiocyanate and then condensing with diethylamine. A solution of 3,5-dinitrobenzoyl chloride (5 mmol) in acetone (50 ml) was added to KSCN (5 mmol) in acetone (25 ml). The mixture was stirred for 30 min at 313 K and then cooled

to room temperature. A solution of diethylamine (5 mmol) was added within 15 min and the mixture was stirred for 2 h. The solid product was purified by recrystallization from ethanol–dichloromethane (1:2).

Crystal data

$C_{12}H_{14}N_4O_5S$
 $M_r = 326.33$
 Monoclinic, $P2_1/c$
 $a = 7.6702$ (13) Å
 $b = 20.626$ (4) Å
 $c = 9.2393$ (16) Å
 $\beta = 99.410$ (3)°
 $V = 1442.0$ (4) Å³

$Z = 4$
 $D_x = 1.503$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 120$ (2) K
 Prism, yellow
 $0.47 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.890$, $T_{\max} = 0.951$

11284 measured reflections
 3488 independent reflections
 2777 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$
 $\theta_{\text{max}} = 28.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.132$
 $S = 1.07$
 3488 reflections
 201 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0731P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C9-H9A\cdots O5^i$	0.99	2.50	3.446 (2)	160
$N3-H3A\cdots O5^i$	0.88	2.30	3.063 (2)	145
$C9-H9B\cdots O2^{ii}$	0.99	2.40	3.184 (2)	136
$N3-H3A\cdots S1^i$	0.88	2.73	3.4318 (16)	138

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

H atoms were found in difference Fourier maps, set to idealized positions ($C-H = 0.95-0.99$ Å and $N-H = 0.88$ Å) and refined as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{methyl C})$. CH_3 groups were allowed to rotate but not to tip.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2002); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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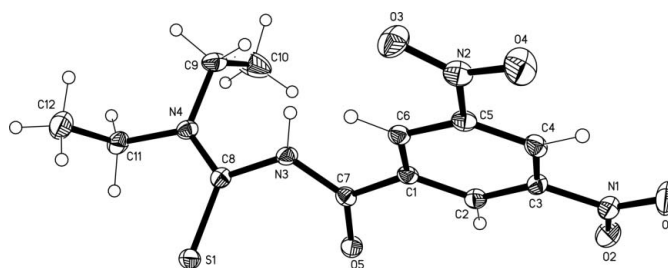


Figure 1
 The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

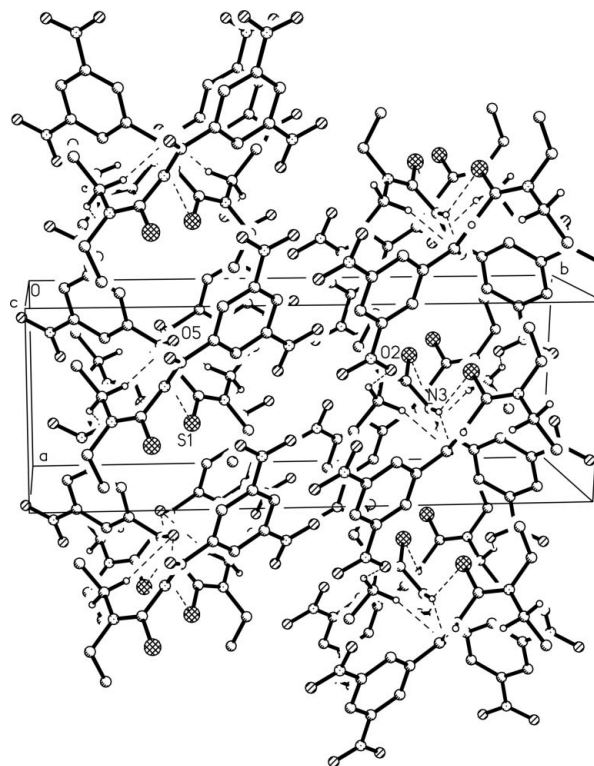


Figure 2
 Crystal packing, viewed along [100], with intermolecular hydrogen bonds indicated as dashed lines. H atoms not involved in the interactions shown have been omitted.

References

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