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3-[4-(3,3-Diethylthioureidocarbonoyl)benzoyl]-1,1-diethylthiourea

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Key indicators

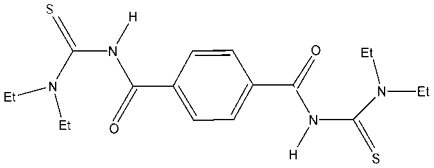
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.090
Data-to-parameter ratio = 16.1

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

The title compound, $C_{18}H_{26}N_4O_2S_2$, lies about a crystallographic inversion centre. The crystal packing shows intermolecular C—H···O and N—H···S interactions, the latter giving rise to the formation of dimers.

Comment

The title compound, (I), is another example of our newly synthesized thiourea derivatives which show interesting complexation capacity.



(I)

The centre of the molecule lies about a crystallographic inversion centre and thus the two thiourea moieties adopt an *anti* position. The planes of the benzoyl ring and the thiourea N_2CS group are almost perpendicular with an angle of 89.0 (1)°. The corresponding torsion angles are N2—C6—C7—C8 = 160.4 (1)°, C6—N2—C5—S1 = 108.3 (1)° and O1—C6—C7—C8 = 154.8 (1)°. Prominent bond lengths are in the expected range, *viz*. C5—S1 = 1.6660 (13) Å, C6—O1 = 1.2185 (16) Å, C5—N2 = 1.4173 (16) Å and C5—N1 = 1.3214 (17) Å and compare well with the related distances of 1,1-diethyl-3-(4-methylbenzoyl)thiourea (Morales *et al.*, 1997) or *N*-benzoyl-*N*′-methyl-*N*′-phenylthiourea (Shanmuga Sundara Raj *et al.*, 1999). In the crystal structure (Fig. 2), molecules form dimers through strong intermolecular N2—H2···S1($1-x$, $-y$, $2-z$) hydrogen bonds, with H···S = 2.37 Å and N—H···S = 172°. An additional intermolecular interaction is C2—H2*B*···O1($x-\frac{1}{2}$, $\frac{1}{2}-y$, $z-\frac{1}{2}$), with H···O = 2.33 Å and C—H···O = 152°. An intramolecular hydrogen bond is formed by C2—H2*A*···N2, with H···N =

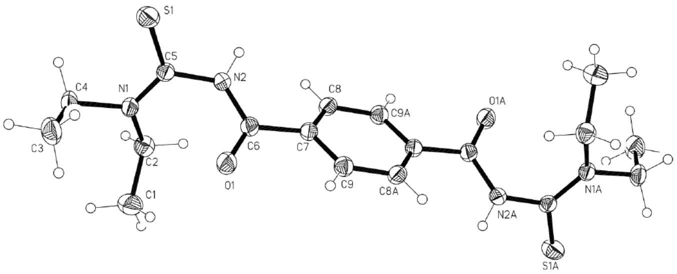


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (*A*) $1-x$, $-y$, $3-z$.]

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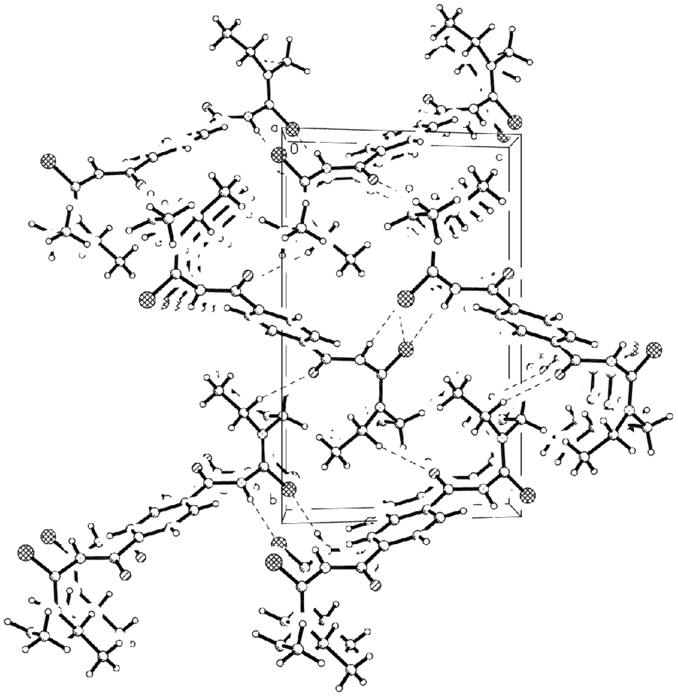

Figure 2
Packing diagram, viewed along [100]. Intermolecular hydrogen bonding is indicated by dashed lines.

2.30 Å and C—H···N = 105°. All these values are normalized for N—H = 1.03 Å and C—H = 1.08 Å.

Experimental

A solution of 2.5 mmol terephthalyl dichloride in 125 ml acetone was added to 5 mmol KSCN in 25 ml of acetone. The mixture was stirred for 30 min at 313 K and then cooled to room temperature. Afterwards a solution of 5 mmol diethylamine in 25 ml acetone was added dropwise with stirring, which was continued for 2 h. The yellowish precipitate was recrystallized from dichloromethane–ethanol (1:1).

Crystal data

$C_{18}H_{26}N_4O_2S_2$
M_r = 394.55
Monoclinic, $P2_1/n$
a = 6.7980 (7) Å
b = 15.2735 (16) Å
c = 10.0275 (10) Å
β = 109.168 (1)°
V = 983.43 (17) Å^3
Z = 2
D_x = 1.332 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3822 reflections
θ = 2.5–28.3°
μ = 0.29 mm^{-1}
T = 173 (2) K
Prism, colourless
0.32 × 0.25 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min}$ = 0.890, $T_{\max}$ = 0.973
5476 measured reflections
1991 independent reflections
1821 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.028
$\theta_{\max}$ = 26.4°
$h = -8 \rightarrow 8$
$k = -19 \rightarrow 17$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.090
S = 1.06
1991 reflections
124 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.2868P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.29 e Å^{-3}
$\Delta\rho_{\min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.6660 (13)	N2—C6	1.3606 (17)
O1—C6	1.2185 (16)	N2—C5	1.4173 (16)
N1—C5	1.3214 (17)	C6—C7	1.4915 (17)
C6—N2—C5	119.59 (10)	O1—C6—N2	122.06 (12)
N1—C5—N2	115.88 (11)	O1—C6—C7	121.05 (12)
N1—C5—S1	125.10 (10)	N2—C6—C7	116.85 (11)
N2—C5—S1	119.02 (9)		

The H atom bonded to N2 was refined freely. Other H atoms were placed at calculated positions, riding on their attached C atoms (C—H = 0.95, 0.98 and 0.99 Å for aromatic, CH_2 and CH_3 H atoms, respectively), with isotropic displacement parameters $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) or $1.5U_{\rm eq}(CH_3)$. CH_3 groups were allowed to rotate, but not to tip.

Data collection: *SMART* (Bruker, 2002); cell refinement: SMART; data reduction: *SAINT* (Bruker, 2002); 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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