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N,*N*-Diisobutyl-*N*′-(2-thienylcarbonyl)thiourea

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Key indicators

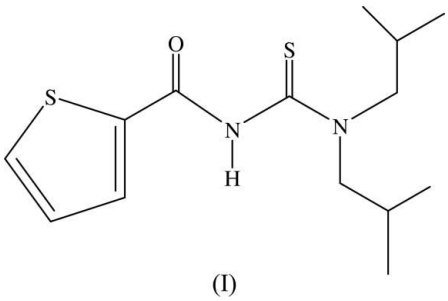
Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.041
wR factor = 0.107
Data-to-parameter ratio = 15.4

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

The title compound, $C_{14}H_{22}N_2OS_2$, contains a disordered isobutyl group. There are no direction-specific interactions between the molecules of the title compound.

Comment

Substituted *N*-acylthioureas have been a subject of investigations, due to their ability to form stable metal complexes, their extraction proprieties and as model compounds in physical chemistry studies (Beyer *et al.*, 1981; Mühl *et al.*, 1986). We report here the structure of *N*,*N*-diisobutyl-*N*′-(2-thienylcarbonyl)thiourea, (I) (Figs. 1 and 2). The main bond lengths are given in Table 1 and are within the ranges obtained for similar compounds (Bailey *et al.*, 1988; Koch *et al.*, 1995; Morales *et al.*, 1997).



(I)

The dihedral angle between the O2/C21/N1 and S1/C6/N2 planes is 39.5 (3)°, while that between the O2/C21/N1 plane and the thiophene ring is 6.8 (4)°

Experimental

The title compound, (I), was prepared by adapting the general procedure described by Douglass & Dains (1934) for other *N*-acyl-

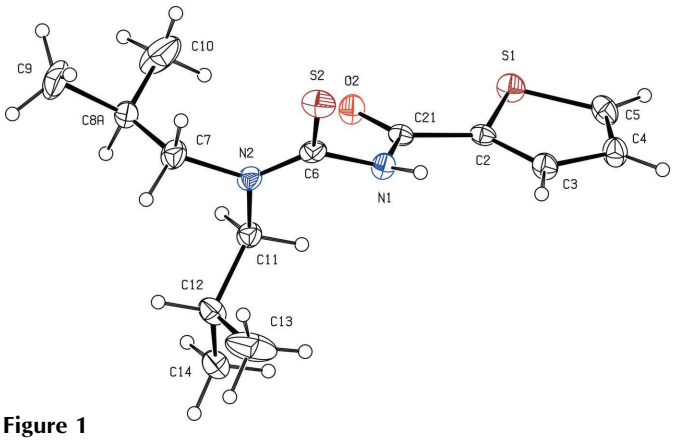


Figure 1
The structure of the major component of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

thioureas. A solution of anhydrous acetone containing thienyl chloride was added to an equimolar acetone–potassium thiocyanate solution. After stirring and boiling the mixture, a solution of equimolar diisobutylamine in acetone was added and kept under reflux for 1 h. The final mixture was poured into a water–ice bath, and crystals were formed. These were filtered and purified by recrystallization from an ethanol/water mixture. The resulting solid product (I) was crystallized from a dichlorometane–methanol (1:1) mixture yielding X-ray quality single crystals (m.p. 413.2 K). Elemental analysis for $C_{14}H_{22}N_2OS_2$ found: C 55.7, H 7.5, N 9.6, S 22.1%; calculated: C 56.34, H 7.43, N 9.4, S 21.5%.

Crystal data

$C_{14}H_{22}N_2OS_2$
M_r = 298.4
Monoclinic, $P2_1/c$
a = 12.0720 (6) Å
b = 8.5169 (7) Å
c = 16.7544 (18) Å
β = 109.243 (6)°
V = 1626.4 (2) Å^3
Z = 4
D_x = 1.215 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 290 (2) K
Prism, yellow
0.3 × 0.2 × 0.1 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
Profile-fitted 2θ/ω scans (Clegg, 1981)
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.888, T_{max} = 0.968
5714 measured reflections
2861 independent reflections
2161 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 25.0°
1 standard reflection
frequency: 80 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.08
2861 reflections
186 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.8882P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected bond lengths (Å).

S2—C6	1.677 (2)	N1—C21	1.383 (3)
S1—C5	1.700 (3)	N1—C6	1.421 (3)
S1—C2	1.724 (2)	N2—C6	1.328 (3)
O2—C21	1.218 (3)		

All the H atoms bonded to C atoms were assigned standard C—H distances [0.93 (aromatic), 0.98 (aliphatic)), 0.96 (methyl) or 0.97 Å (CH_2), with U_{iso}(H) = kU_{eq}(C), where k = 1.5 for methyl groups and 1.2 for other H atoms bonded to C atoms, and N—H = 0.83 Å, with U_{iso}(H) = 1.2U_{eq}(N)]. The central atom of the isobutyl group containing atoms C7, C9 and C10 is disordered over two sites, *viz*. C8*A* and C8*B*. H atoms were calculated for atoms C7, C8, C9 and C10 on the basis of this disorder. Those for the methyl atoms C9 and C10 were calculated as six equally spaced half-occupancy hydrogens. The refined values of site-occupancy factors for the disordered groups attached to C8*A* and C8*B* are 0.642 (14) and 0.458 (14), respectively.

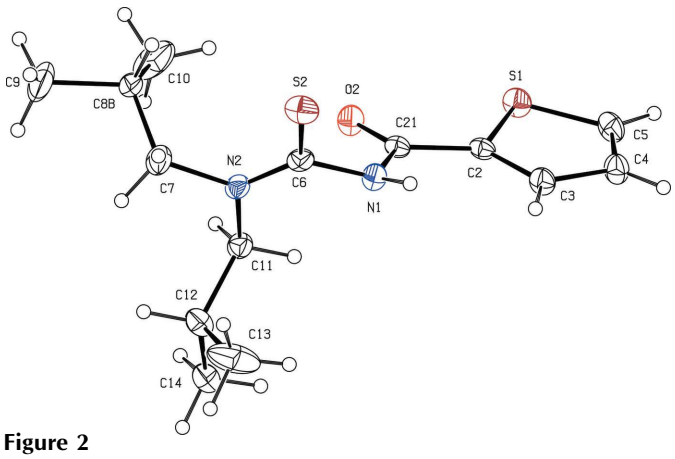


Figure 2
The structure of the minor component of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *IPDS Software* (Stoe & Cie, 1996); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON*, (Spek, 2003); software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993).

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