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1-{(2*E*)-2-[(Aminocarbonothioyl)hydrazono]-2-(3-mesityl-3-methylcyclobutyl)ethyl}-pyrrolidine-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.110
Data-to-parameter ratio = 14.6

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

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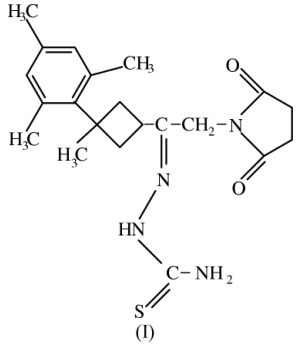
The molecule of the title compound, $C_{21}H_{28}N_4O_2S$, contains three rings, namely cyclobutane, pyrrolidine and benzene. In the wedge-shaped cyclobutane ring, the maximum deviation from planarity is 0.151 (3) Å. The molecules are linked by N—H···S and N—H···O intermolecular hydrogen bonds.

Comment

The title compound is one of a type also known as N4-substituted thiosemicarbazones. Thiosemicarbazone (TSC) derivatives are a class of compounds that possess a range of biological properties: antitumour, antibacterial, antiviral, antimalarial and antifungal activities have been reported (West *et al.*, 1991; Klayman *et al.*, 1979; Bermejo *et al.*, 1999). The biological activities of thiosemicarbazones are considered to be due to their ability to form chelates with metals. The biological activities of the metal complexes differ from those of either the ligands or the metal ions, and increased or decreased biological activities have been reported for several transition metal complexes, such as Cu and Ni complexes (Liberta *et al.*, 1992; West *et al.*, 1991). Therefore, the crystal structure determination of the title compound, (I), has been carried out and the results are presented here.



(I)

The molecule of (I) (Fig. 1) contains three rings, namely a cyclobutane ring (C8–C11), a pyrrolidine ring (N4/C4–C7) and a benzene ring (C13–C18). The thiosemicarbazone moiety is planar, forming dihedral angles of 64.5 (1)° with the cyclobutane ring, 83.0 (1)° with the pyrrolidine ring and 89.8 (1)° with the benzene ring. The dihedral angle formed by the cyclobutane and benzene rings is 37.3 (1)°. The bond distances and angles in the thiosemicarbazone moiety of (I) agree with the literature values (Beraldo *et al.*, 2003). Selected bond lengths and angles for (I) are listed in Table 1.

Intermolecular N—H···O and N—H···S hydrogen bonds are present in the crystal structure of (I) and details of these are given in Table 2.

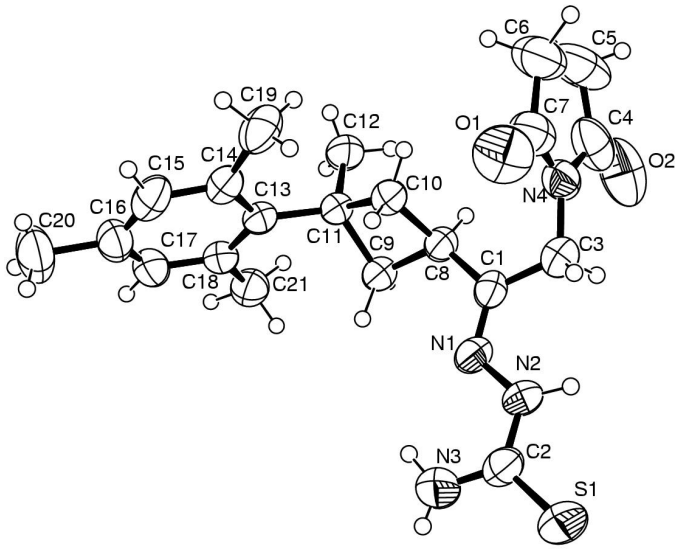


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

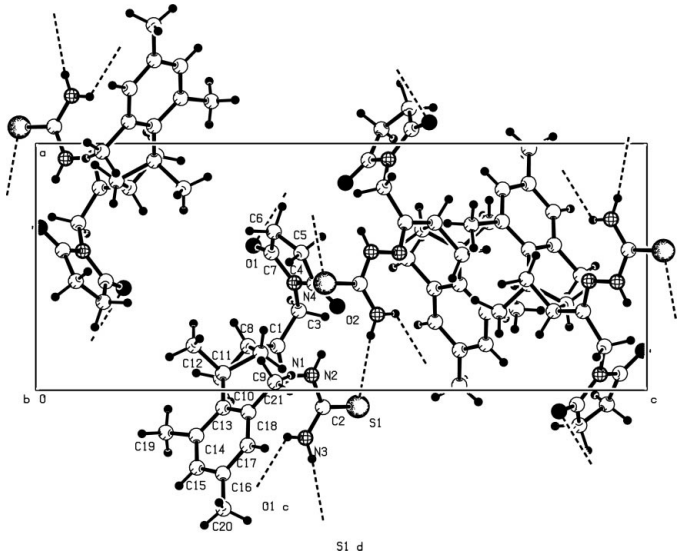


Figure 2
The molecular packing in (I), viewed down the *b* axis.

Experimental

A mixture of 1-methyl-1-mesityl-3-(2-chloro-1-oxoethyl)cyclobutane (3.344 g, 12.5 mmol), succinimide (1.239 g, 12.5 mmol) and K_2CO_3 (0.864 g, 6.25 mmol) in ethanol (50 ml) was refluxed for 16 h with stirring. Completion of the reaction was easily observed by monitoring the IR frequency of the $-CH_2-Cl$ group of the α-haloketone. Subsequently, thiosemicarbazide (1.139 g, 12.5 mmol) was added gradually and the mixture refluxed for a further 24 h. After cooling to room temperature and adding water (100 ml), the solid which formed was filtered off, washed several times with cold ethanol and water and recrystallized from diethyl ether (yield 4.14 g, 83%; m.p. 288 K). Spectroscopic analysis: IR (ν, cm^{-1}): 3290 and 3440 ($-NH_2$), 2978–2927 (aliphatic C—H), 1714 (C═O), 1589 (C═N), 1084 (C═S); ^{1}H NMR ($CDCl_3$, TMS, δ, p.p.m.): 1.56 (*s*, 3H, CH_3), 2.20 (*s*, 9H, CH_3 on mesitylene), 2.43–2.65 (*m*, 4H, $-CH_2-$ in cyclobutane), 2.80 (*s*, 4H, $-CH_2-$ on succinimide), 3.38 (*quint*, J = 8.9 Hz, 1H, >CH– in cyclobutane ring), 4.17 (*s*, 2H, $-CH_2-N$), 6.41 (*s*, 1H, –NH– from $-NH_2$), 6.75 (*s*, 2H, aromatics on mesitylene),7.07 (*s*, 1H, –NH– from $-NH_2$), 10.20 (*s*, 1H, –NH–); ^{13}C NMR ($CDCl_3$, TMS, δ, p.p.m.): 176.30 (C1), 28.70 (C2), 40.70 (C3), 149.30 (C4), 34.20 (C5), 34.90 (C6), 39.50 (C7), 27.70 (C8), 134.30 (C9), 129.70 (C10), 133.45 (C11), 142.80 (C12), 20.70 (C13), 23.20 (C14), 178.70 (C15). Analysis calculated for $C_{21}H_{28}N_4O_2S$: C 62.97, H 7.05, N 13.99, S 8.01%; found: C 63.17, H 7.24, N 14.42, S 7.88%.

Crystal data

$C_{21}H_{28}N_4O_2S$
M_r = 400.53
Orthorhombic, $P2_12_12_1$
a = 8.8720 (7) Å
b = 10.8100 (11) Å
c = 21.9284 (19) Å
V = 2103.1 (3) Å^3
Z = 4
D_x = 1.265 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4921 reflections
θ = 1.2–29.8°
μ = 0.18 mm^{-1}
T = 293 (2) K
Irregular, colourless
0.52 × 0.34 × 0.22 mm

Data collection

Stoe IPDS 2 diffractometer
φ scans
Absorption correction: by integration (*X-RED*; Stoe & Cie, 2001)
T_{min} = 0.936, T_{max} = 0.971
9857 measured reflections
3703 independent reflections
2270 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 25.0°
$h = -10 \rightarrow 9$
$k = -12 \rightarrow 11$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.110
S = 0.88
3703 reflections
254 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0032 (11)
Absolute structure: Flack (1983), 1571 Friedel pairs
Flack parameter = 0.00 (13)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.198 (5)	C1—N1	1.281 (4)
C7—N4	1.374 (5)	N2—C2	1.343 (4)
N4—C4	1.384 (5)	C2—N3	1.317 (5)
C4—O2	1.194 (5)	C2—S1	1.672 (3)
C1—N1—N2	116.7 (3)	N3—C2—S1	123.0 (3)
C2—N2—N1	119.3 (3)	N2—C2—S1	119.5 (3)
N3—C2—N2	117.5 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3D···S1^{i}	0.86	2.87	3.725 (3)	173
N3—H3C···O1^{ii}	0.86	2.53	3.114 (5)	126

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

All H atoms were positioned geometrically and refined as riding, with C—H= 0.93–0.98 Å and N—H = 0.86 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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