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3-Butyl-4-(3-methyl-3-phenylcyclobut-1-yl)-1,3-thiazole-2(3*H*)-thione

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Key indicators

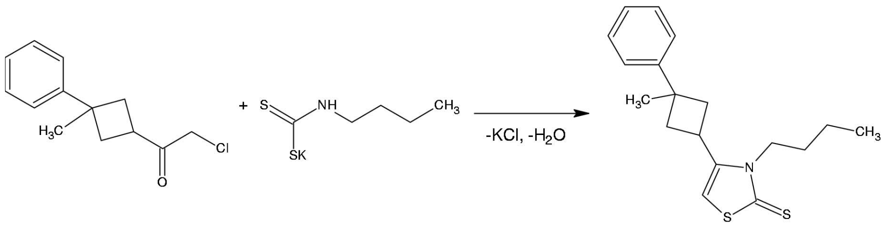
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.070
Data-to-parameter ratio = 14.8

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

In the title compound, $C_{18}H_{23}NS_2$, the bond lengths and angles are unexceptional. The butyl chain is in a fully extended conformation. The dihedral angle between the thiazole and phenyl rings is 87.21 (2)°. The crystal structure is stabilized by one weak intramolecular C—H···S hydrogen bond.

Comment

1,3-Thiazole-2(3*H*)-thiones have found many uses in recent years, in such diverse fields as photography, agrochemistry, and radiochemistry; in agrochemistry in particular, they are potential plant-protecting compounds, *e.g.* as fungicides and insecticides (Ahmedzade, Çukurovalı & Koparır, 2003). It has been reported that several thiazoles and thiazolidin-4-ones are of biological importance, especially as antimetabolites and schistosomicides (El-Zahar *et al.*, 1994). Various thiazole derivatives show herbicidal, anti-inflammatory, antimicrobial and antiparasite activity (Koparır *et al.*, 2004; Ahmedzade, Çukurovalı & Koparır, 2003), and also liquid crystal properties (Coghi *et al.*, 1976).



(I)

The bond lengths and angles of the thiazolethione system of the title compound, (I), are unexceptional and agree well with previously reported values (Hartung *et al.*, 2003; Kanagapushpam & Venkatesan, 1987). Likewise, the geometric parameters of the cyclobutane ring are in accord with literature values (Özdemir *et al.*, 2004; Yüksektepe *et al.*, 2004). The butyl chain is in a fully extended conformation, the N1—C15—C16—C17 and C15—C16—C17—C18 torsion angles being 173.3 (1) and 175.1 (2)°, respectively.

The thiazolethione system is essentially planar, the maximum deviation from planarity being 0.006 (1) Å for atom S2. The dihedral angle between the thiazole and phenyl rings is 87.21 (2)°. The cyclobutane ring is puckered, the C10/C11/C9 plane forming a dihedral angle of 24.37 (3)° with the C9/C7/C10 plane. This compares well with the value of 23.5 (4)° reported for a hexafluorocyclobutane (Swenson *et al.*, 1997).

The crystal structure is stabilized by one weak intramolecular C—H···S hydrogen bond (Table 1). The C···S distance of 3.132 (2) Å is longer than that observed in a similar thiazoline compound [2.69 (6) Å; Aydın *et al.*, 1999].

Experimental

2-Chloro-1-(3-methyl-3-phenylcyclobutyl)ethan-1-one (4.45 g, 0.02 mol) was added in portions to a stirred slurry containing potassium benzylcarbamodithioate (4.4 g, 0.02 mol) in ethanol (40 ml). After stirring for 50 min, the temperature had risen from 293 to 313 K. The stirred reaction mixture was heated at reflux (351–353 K) for 4 h and then allowed to cool. Stirring was maintained at 298–303 K for 24 h. After cooling to 278 K, water (60 ml) was added. The solid was collected by filtration, washed with water and dried in air at 298–303 K. The elemental analysis, IR and NMR spectroscopic data of the title compound have already been published (Ahmedzade, Kırılmış *et al.*, 2003).

Crystal data

$C_{18}H_{23}NS_2$
$M_r = 317.51$
Orthorhombic, $P2_12_12_1$
$a = 9.908$ (1) Å
$b = 10.442$ (1) Å
$c = 16.603$ (2) Å
$V = 1717.7$ (3) Å^3
$Z = 4$
$D_x = 1.228$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 313 reflections
$\theta = 6.0$–$20.0°$
$\mu = 0.30$ mm^{-1}
$T = 100$ (2) K
Prism, colourless
$0.30 \times 0.28 \times 0.23$ mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* (Bruke, 2002)
$T_{\min} = 0.913$, $T_{\max} = 0.932$
19 922 measured reflections
3833 independent reflections
3274 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.070$
$S = 1.02$
3833 reflections
259 parameters
Only positions of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.237P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Absolute structure: Flack (1983), 1582 Friedel pairs
Flack parameter = 0.04 (6)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15A···S2	0.99 (3)	2.78 (2)	3.132 (2)	102 (2)

H atoms were refined in x, y, z, while their $U_{\rm iso}$ values were fixed at 0.040 Å^2.

Data collection: *COLLECT* (Bruker, 2002); cell refinement: *EVALCCD* (Bruker, 2002); data reduction: *EVALCCD*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

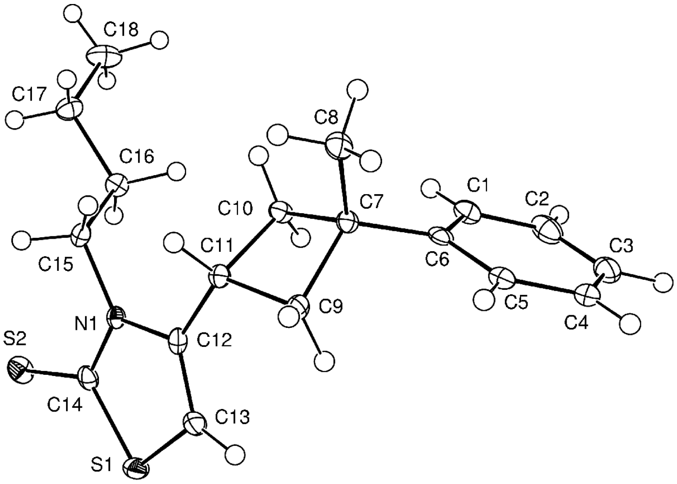


Figure 1
ORTEP-3 plot (Farrugia, 1997) of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

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