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Key indicators

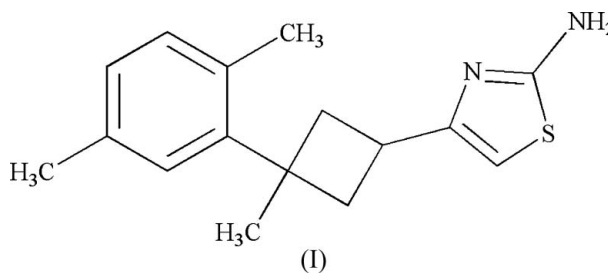
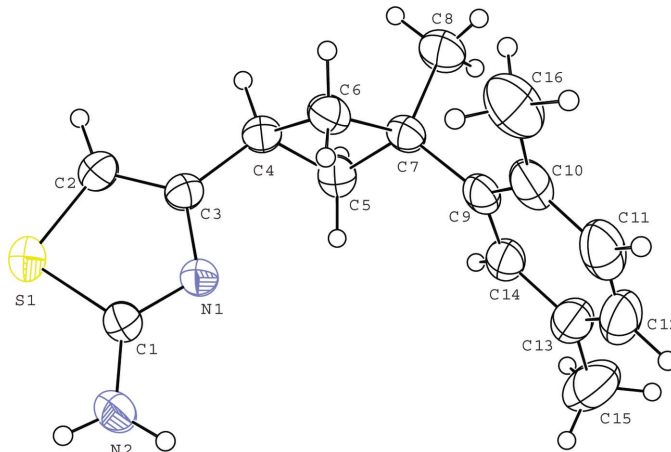
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.039
 wR factor = 0.100
Data-to-parameter ratio = 19.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.2-Amino-4-(3-methyl-3-*p*-xylylcyclobutyl)-
1,3-thiazoleIn the title compound, $\text{C}_{16}\text{H}_{20}\text{N}_2\text{S}$, an intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond and an $\text{N}-\text{H}\cdots\pi$ interaction link two molecules, forming a dimer.

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Comment

Various thiazole derivatives show herbicidal, anti-inflammatory, antimicrobial and antiparasitic activity (Koparir *et al.*, 2004; Ahmedzade *et al.*, 2003), and also liquid crystal properties (Coghi *et al.*, 1976). Aminothiazoles have been extensively studied for a range of biological and industrial applications (Lynch *et al.*, 1999; Toplak *et al.*, 2003). 2-Amino-1,3-thiazole, the structure of which was reported by Caranoni & Reboul (1982), is itself listed as a thyroid inhibitor (Merck, 2001).The dihedral angle between the thiazole and benzene rings is $81.88(9)^\circ$, comparable to $87.21(2)^\circ$ and $89.70(2)^\circ$ reported for 3-butyl-4-(3-methyl-3-phenylcyclobutyl)-1,3-thiazole-**Figure 1**
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

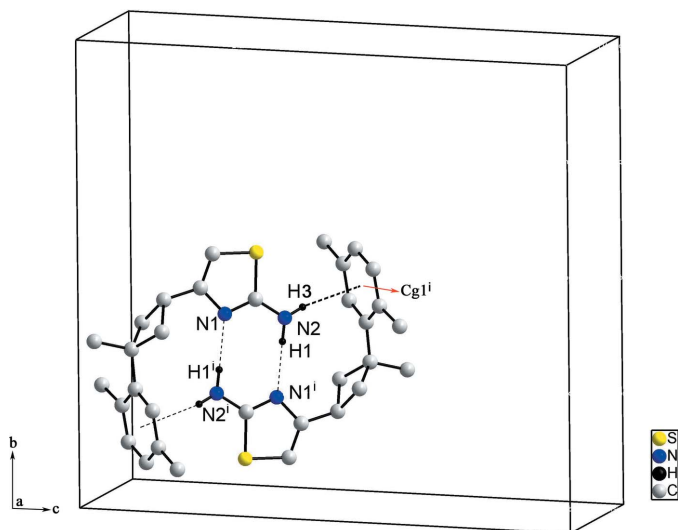


Figure 2

A view of the hydrogen-bonding interactions (dashed lines) in (I). For the sake of clarity, H atoms bonded to C atoms have been omitted. Cg1 is the centroid of the C9–C14 benzene ring [symmetry code: (i) $\frac{1}{3} - x, \frac{2}{3} - y, \frac{2}{3} - z$].

2(3*H*)-thione (Kirilmış *et al.*, 2005*a*) and 3-cyclohexyl-4-(3-methyl-3-phenylcyclobutyl)-1,3-thiazole-2(3*H*)-thione (Kirilmış *et al.*, 2005*b*), respectively. The cyclobutane ring is puckered; the C5/C4/C6 plane forms a dihedral angle of 20.8 (2)^o with the C6/C7/C5 plane. This compares well with the values of 24.37^o, 23.82 (3)^o and 23.5 (4)^o reported for the above-mentioned compounds (Kirilmış *et al.*, 2005*a,b*) and hexafluorocyclobutane (Swenson *et al.*, 1997), respectively. An intermolecular N2–H1···N1ⁱ hydrogen bond (symmetry code as in Table 2) links the molecules, forming a hydrogen-bonded dimer of graph-set motif $R_2^2(8)$ (Fig. 2). Of greater interest is an intermolecular N–H··· π interaction between the amino group and the benzene ring in the dimer (Table 2). As a result of these interactions, as well as van der Waals interactions, the structure exhibits hexagonal cavities of diameter 3.774 Å along the *c* axis (Fig. 3).

Experimental

To a solution of thiourea (0.76 g, 10 mmol) in absolute ethanol (50 ml), a solution of 3-(2-chloro-1-oxoethyl)-1-methyl-1-*p*-xylylcyclobutane (2.50 g, 10 mmol) in absolute ethanol (20 ml) was added dropwise at 323–327 K with continuous stirring. By monitoring the IR frequency of the carbonyl group of 3-(2-chloro-1-oxoethyl)-1-methyl-1-*p*-xylylcyclobutane, completion of the reaction was easily seen. After cooling to room temperature, the solution was made alkaline with an aqueous solution of NH₃ (5%), causing the separation of dark-green crystals of (I) from the reaction mixture. The precipitate was filtered off, washed with an aqueous ammonia solution and water several times, dried in air and recrystallized from water–ethanol (1:4) by slow evaporation (yield 76%; m.p. 407.5 K). Characteristic ¹H NMR shifts (CDCl₃, p.p.m.) are at 1.54 (*s*, 3H, –CH₃ on cyclobutane), 2.22 (*s*, 3H, *o*-CH₃ of *p*-xylyl), 2.33 (*s*, 3H, *m*-CH₃ of *p*-xylyl), 2.48 (*m*, 2H, –CH₂–), 2.59 (*m*, 2H, –CH₂–), 3.43 (*q*, *J* = 9.2, 1H, >CH–), 5.55 (*brs*, 2H, NH₂), 5.96 (*s*, 1H, aromatic in thiazole ring), 6.89 (*s*, 1H, aromatic), 7.03 (*m*, 2H, aromatic). Characteristic ¹³C NMR shifts

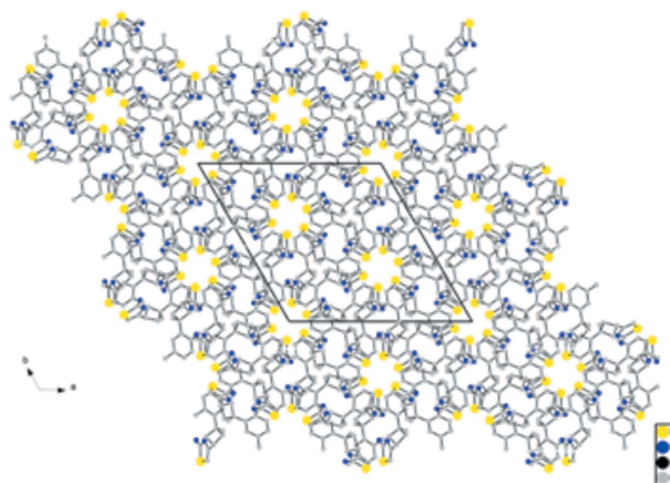


Figure 3

A view of the cavities along the *c* axis. H atoms have been omitted.

(CDCl₃, p.p.m.) are at 169.18, 155.84, 149.80, 135.57, 132.12, 131.43, 127.11, 126.85, 100.39, 41.89, 39.46, 31.09, 28.05, 21.29 and 19.71.

Crystal data

C₁₆H₂₀N₂S
M_r = 272.40
 Trigonal, $R\bar{3}$
a = 20.7083 (11) Å
c = 18.2954 (10) Å
V = 6794.6 (6) Å³
Z = 18
D_x = 1.198 Mg m^{−3}

Mo *K*α radiation
 Cell parameters from 37485 reflections
 θ = 1.6–28.0^o
 μ = 0.20 mm^{−1}
T = 293 (2) K
 Prism, pale yellow
 0.57 × 0.48 × 0.38 mm

Data collection

Stoe IPDS-2 diffractometer
 ω scans
 Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.900, *T_{max}* = 0.944
 37485 measured reflections
 3557 independent reflections

2851 reflections with *I* > 2σ(*I*)
R_{int} = 0.068
 θ_{\max} = 27.8^o
h = −26 → 27
k = −27 → 27
l = −23 → 23

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.039
wR(*F*²) = 0.100
S = 1.05
 3557 reflections
 182 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 3.0488P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.16 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1–C2	1.7251 (16)	N1–C3	1.3890 (16)
S1–C1	1.7424 (14)	C2–C3	1.3436 (19)
N1–C1	1.2998 (17)	C3–C4	1.4914 (19)
N1–C1–N2	124.34 (13)	C3–C4–C5	117.36 (12)
N1–C1–S1	114.56 (10)	C9–C7–C5	116.10 (12)
C2–C3–C4–C6	124.11 (15)	C2–C3–C4–C5	−133.16 (15)
N1–C3–C4–C6	−55.34 (17)	N1–C3–C4–C5	47.39 (18)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H1\cdots N1^i$	0.91 (2)	2.15 (2)	3.0540 (19)	180 (2)
$N2-H3\cdots Cg1^i$	0.83 (2)	2.45 (1)	3.307 (2)	167 (2)

Symmetry code: (i) $-x + \frac{1}{3}, -y + \frac{2}{3}, -z + \frac{2}{3}$. Cg1 is the centroid of the C9–C14 benzene ring

All H atoms, except for H1 and H3, were placed in calculated positions, with C–H distances in the range of 0.93–0.96 Å. $U_{iso}(H)$ values were constrained to be 1.2 and 1.5 times U_{eq} of the carrier atom. Atoms H1 and H3 were located in a difference map and refined freely.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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