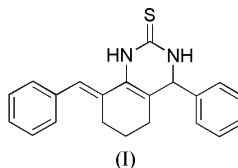


(8*E*)-8-Benzylidene-3,4,5,6,7,8-hexahydro-4-phenylquinazoline-2(1*H*)-thione**Xiu-Ling Zhang^{a*}** and
Zu-Pei Liang^b^aDepartment of Chemistry, Dezhou University, Dezhou 253011, People's Republic of China, and ^bCollege of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of ChinaCorrespondence e-mail:
zupeiliang@yahoo.com.cn**Key indicators**Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
Disorder in main residue
R factor = 0.048
wR factor = 0.129
Data-to-parameter ratio = 15.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The asymmetric unit of the title compound, $\text{C}_{21}\text{H}_{20}\text{N}_2\text{S}$, comprises two independent molecules, which differ in the conformations of the cyclohexene and dihydropyrimidine rings, and in the orientations of the benzylidene moieties. The crystal structure is stabilized by weak $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds.

Comment

Quinazoline and its synthetic analogues have been found to exhibit a broad spectrum of activity such as antibacterial (Bedi *et al.*, 2004), antimicrobial (Trivedi *et al.*, 1993), analgesic (Ram *et al.*, 1990) and anti-helminthic (Gupta *et al.*, 1998). Thus there has been considerable interest in the chemistry of quinazoline and its synthetic analogues in recent years. In this paper, the structure of the title compound, (I), a quinazoline derivative, is reported.



The asymmetric unit of (I) (Fig. 1) consists of two independent molecules, which differ in the conformations of the cyclohexene and dihydropyrimidine rings. The orientations of the benzylidene moieties are also different in the two molecules. In one of the molecules, the dihydropyrimidine ring [defined by atoms N1, N2, C1–C4] adopts a half-chair conformation, whereas in the other molecule the dihydropyrimidine ring [defined by atoms N3, N4, C22–C24] is planar within $0.034(3) \text{ \AA}$. The planar pyrimidine ring forms a dihedral angle of $81.0(2)^\circ$ with the attached phenyl ring. The cyclohexene ring containing atoms C24 to C29 adopts a half-chair conformation. The other cyclohexene ring is disordered; both the major and minor conformers adopt envelope conformations. The $\text{C}5-\text{C}15-\text{C}16-\text{C}21$ [$-22.0(5)^\circ$] and $\text{C}26-\text{C}36-\text{C}37-\text{C}38$ [$-44.6(4)^\circ$] torsion angles indicate the different orientation of benzylidene phenyl rings in the two independent molecules. Selected geometric parameters are listed in Table 1. The crystal structure is stabilized by weak $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds (Fig. 2 and Table 2).

Experimental

A mixture of (2*E*,6*E*)-2,6-dibenzylidenecyclohexanone (0.02 mol) and thiourea (0.02 mol) was added to a solution of KOH (2 g) in ethanol (200 ml), and the mixture was refluxed for 3 h. After cooling

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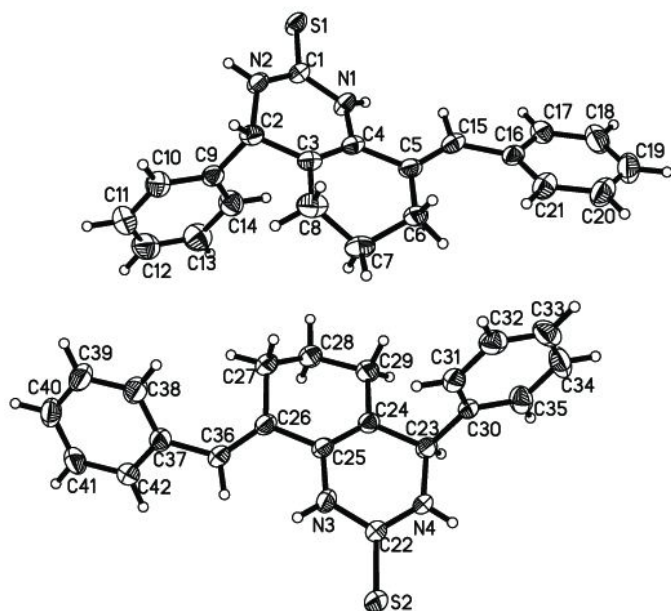


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level. Only the major conformer of the disordered cyclohexene ring is shown.

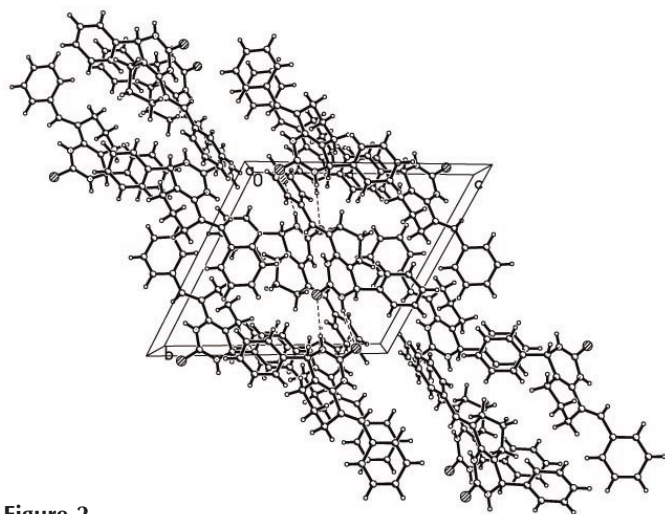


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

and filtration, the title compound was recrystallized from ethanol (m.p. 469–470 K). 20 mg of the compound was dissolved in 15 ml of a mixed solvent containing ethyl acetate (5 ml) and methanol (10 ml). The solution was allowed to evaporate at room temperature and colourless single crystals were formed after 12 d.

Crystal data

$C_{21}H_{20}N_2S$	$Z = 4$
$M_r = 332.45$	$D_x = 1.243 \text{ Mg m}^{-3}$
Triclinic, $P1$	Mo $K\alpha$ radiation
$a = 10.472 (3) \text{ \AA}$	Cell parameters from 771 reflections
$b = 13.526 (4) \text{ \AA}$	$\theta = 3.2\text{--}25.1^\circ$
$c = 14.963 (5) \text{ \AA}$	$\mu = 0.19 \text{ mm}^{-1}$
$\alpha = 113.440 (5)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 100.681 (6)^\circ$	Block, colourless
$\gamma = 104.926 (6)^\circ$	$0.32 \times 0.30 \times 0.24 \text{ mm}$
$V = 1777.2 (10) \text{ \AA}^3$	

Data collection

Bruker SMART1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.928$, $T_{\max} = 0.956$
 10378 measured reflections

7205 independent reflections
 3846 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -11 \rightarrow 13$
 $k = -16 \rightarrow 15$
 $l = -14 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.129$
 $S = 1.00$
 7205 reflections
 477 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1289P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—C1	1.696 (3)	S2—C22	1.679 (3)
N1—C1	1.347 (3)	N3—C22	1.350 (3)
N1—C4	1.401 (3)	N3—C25	1.398 (3)
N2—C1	1.318 (3)	N4—C22	1.319 (3)
N2—C2	1.467 (3)	N4—C23	1.471 (3)
C2—C3	1.507 (3)	C23—C24	1.506 (3)
C3—C4	1.335 (3)	C24—C25	1.334 (3)
C5—C15	1.327 (3)	C26—C36	1.334 (3)
C1—N1—C4	123.3 (2)	C22—N3—C25	124.0 (2)
C1—N2—C2	124.3 (2)	C22—N4—C23	128.0 (2)
N2—C1—N1	117.4 (2)	N4—C22—N3	116.3 (2)
N2—C2—C3	110.0 (2)	N4—C23—C24	109.8 (2)
C4—C3—C2	120.2 (2)	C25—C24—C23	121.8 (2)
C3—C4—N1	119.6 (2)	C24—C25—N3	119.8 (2)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...S1 ⁱ	0.85 (3)	2.57 (3)	3.396 (3)	163 (2)
N2—H2...S2 ⁱⁱ	0.85 (3)	2.52 (3)	3.352 (3)	167 (2)
N4—H4...S1 ⁱⁱⁱ	0.80 (3)	2.56 (3)	3.342 (3)	165 (3)

Symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $x - 1, y - 1, z$; (iii) $1 + x, 1 + y, z$.

Atoms C6, C7 and C8 of the cyclohexene ring were found to be disordered. The occupancies of the disordered positions C6/C6', C7/C7' and C8/C8' were refined to 0.594 (12)/0.406 (12). The C—C bond distances involving the disordered atoms were restrained to be equal to 1.50 (1) or 1.52 (1) \AA ; the U_{ij} components of the disordered atoms were approximated to isotropic behaviour. The N-bound H atoms were located in a difference Fourier map and refined isotropically; the N—H distances lie in the range 0.81 (2)–0.85 (3) \AA . All other H atoms were positioned geometrically [C—H = 0.93–0.98 \AA] and allowed to ride on their attached atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

SHELXTL (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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