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11-Chloro-5,7-dihydro-6*H*-benzothiazolo[2,3-*b*]-benzo[*h*]quinazoline

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.102
Data-to-parameter ratio = 15.9

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

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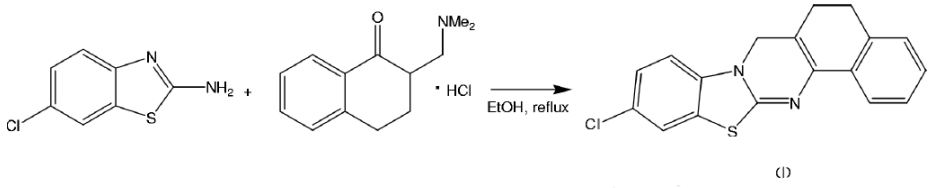
Two independent molecules of the title compound, $C_{18}H_{13}ClN_2S$, exibit a noticeable difference in the conformation of the cyclohexadiene fragment, which shows much more pronounced deviations from planarity in one of the molecules than in the other.

Comment

Benzothiazole and its fused derivatives have shown a wide range of remarkable activities, *viz*. fungicidal, analgesic, anti-inflammatory, anticonvulsant, anaesthetic and pesticide (Di Braccio *et al.*, 1986; Evans, 1974; Baetz, 1973; Mehra *et al.*, 1980; Bartovic *et al.*, 1995; Khan & Rastogi, 1991).

We have used different 2-aminobenzothiazole derivatives with Mannich bases to prepare similar fused derivatives (Quiroga *et al.*, 1992, 1994, 1999; Quiroga, Insuasty, Hernández *et al.*, 1998; Quiroga, Insuasty, Cruz *et al.*, 1998).



(I)

Here we report the structure of 11-chloro-5,7-dihydro-6*H*-benzothiazolo[2,3-*b*]benzo[*h*]quinazoline, (I), prepared by reaction of 2-amino-6-chlorobenzothiazole with 2-dimethylaminomethylenetetralone hydrochloride. Two independent molecules (Figs. 1 and 2) in the asymmetric unit of (I) differ in the degree of non-planarity of the cyclohexadiene rings (C10*A*—C16*B*—C16*A*—C12*A*—C112—C111 and C21*A*—

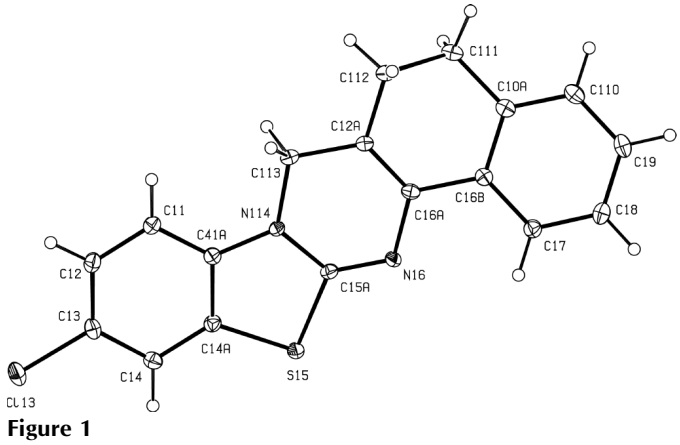


Figure 1
View of molecule *A* of (I) showing the numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as circles of arbitrary radii.

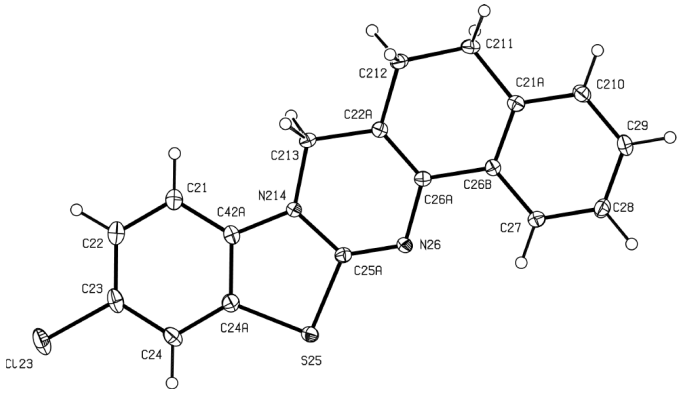


Figure 2
View of molecule *B* of (I) showing the numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as circles of arbitrary radii.

C26*B*—C26*A*—C22*A*—C212—C211 in molecules *A* and *B*, respectively). As one can see from the torsion angles (Table 1), the deviations from planarity in molecule *A* are much more pronounced than in molecule *B*.

Experimental

A solution of 2-amino-6-chlorobenzothiazole (0.5 mmol) and 2-dimethylaminomethylenetetralone hydrochloride (1 mmol) in absolute ethanol (15 ml) was heated to reflux for 30 min. The title compound was isolated by cooling, followed by filtration, washing with ethanol, drying and recrystallization from ethanol. The compound was obtained as yellow crystals; m.p 452 K, yield 70%.

Crystal data

$C_{18}H_{13}ClN_2S$
M_r = 324.82
Triclinic, $P\overline{1}$
a = 7.2637 (3) Å
b = 14.4294 (5) Å
c = 15.1081 (5) Å
α = 70.368 (3)°
β = 78.550 (2)°
γ = 89.0800 (14)°
V = 1459.56 (9) Å^3
Z = 4
D_x = 1.478 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20568 reflections
θ = 2.9–27.5°
μ = 0.40 mm^{-1}
T = 120 (1) K
Block, orange
0.20 × 0.10 × 0.10 mm

Data collection

Kappa–CCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.924, T_{max} = 0.961
20 548 measured reflections
6309 independent reflections
4470 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 27.4°
$h = -9 \rightarrow 9$
$k = -18 \rightarrow 18$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.102
S = 1.03
6309 reflections
397 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.0093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected torsion angles (°).

C12*A*—C16*A*—C16*B*—C10*A*	−16.9 (3)	C22*A*—C26*A*—C26*B*—C21*A*	−7.1 (3)
C16*A*—C16*B*—C10*A*—C111	−1.6 (2)	C26*A*—C26*B*—C21*A*—C211	−5.7 (2)
C16*B*—C10*A*—C111—C112	32.9 (2)	C26*B*—C21*A*—C211—C212	26.3 (2)
C10*A*—C111—C112—C12*A*	−45.8 (2)	C21*A*—C211—C212—C22*A*	−33.6 (2)
C16*B*—C16*A*—C12*A*—C112	1.0 (3)	C26*B*—C26*A*—C22*A*—C212	−2.5 (3)
C111—C112—C12*A*—C16*A*	30.7 (2)	C211—C212—C22*A*—C26*A*	23.1 (3)

H atoms were taken into account in the riding-motion approximation with U_{iso} equal to 1.2U_{eq} of the corresonding carrier atom.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton. The authors thank the staff for all their help and advice.

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