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2,3,4,5-Tetrahydro-1,3-thiazepino[3,2-*a*]-[1,3]benzimidazole

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.001 Å
R factor = 0.052
wR factor = 0.091
Data-to-parameter ratio = 11.6

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

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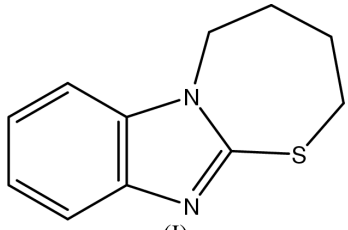
The structure of the title compound, $C_{11}H_{12}N_2S$, established by an X-ray crystallographic study, shows that the molecule is composed of three cycles: bicyclic benzimidazole and the thiazepine ring which is bent.

Comment

Thiazepinobenzimidazole derivatives constitute an interesting series of heterocyclic compounds, particularly for their biological activities (Nawrocka & Zimecki, 1998; Taniguchi *et al.*, 1993; Kuehler *et al.*, 1998; Tabata *et al.*, 1995; Pedini *et al.*, 1994; Piras *et al.*, 1993; Nukaya *et al.*, 1991). We report here the preparation and crystal structure determination of the title compound, (I).



(I)

Selected bond distances and angles are given in Table 1. The molecule is composed of three rings, *viz*. the benzimidazole system and a thiazepine ring. The benzimidazole system is planar (r.m.s. deviation: 0.0136 Å) and the thiazepine seven-membered ring is composed of three planar fragments: S1/C2/N10/C11, which is coplanar with the benzimidazole and at an angle of 118.9 (5)° to the S1/C11/C12/C14 fragment, which is, in turn, at an angle of 121.1 (4)° to the C12/C13/C14 triangle. According to Cremer & Pople (1975), the seven-membered ring conformation can be described on the basis of the total puckering amplitude Q_T = 0.872 (1) Å and the asymmetry parameters that are indicative of a local pseudo-twofold axis running through C13 and the midpoint of the C2—N10 bond.

Experimental

A solution of benzimidazole-2-thione (0.007 mol) and 1,4-dibromobutane (0.014 mol) in 50 ml of saturated aqueous solution of sodium bicarbonate and 50 ml of 2-propanol, was heated under reflux for 1 h. After isolation of 1,4-bis(2-mercaptobenzimidazolyl)butane, the reaction product was obtained by removing the 2-propanol and then extracting the residue with chloroform. Removal of chloroform gave the title compound, yield: 40%, m.p. 403–407 K. Crystals were obtained by evaporation of an ethanol solution at room temperature.

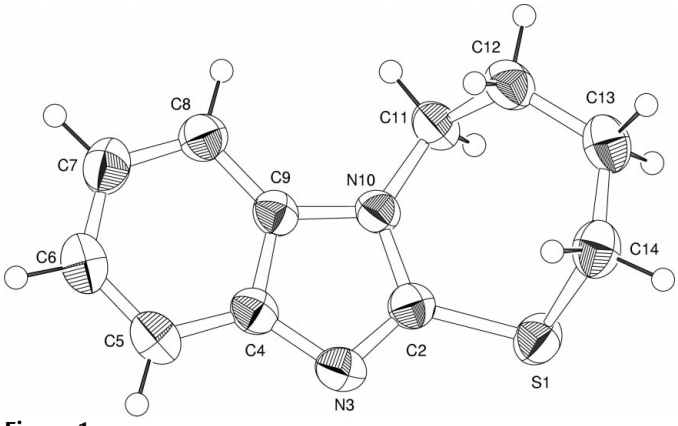


Figure 1
Perspective view of the title molecule showing the labelling of the atoms, with displacement ellipsoids at the 50% probability level.

Crystal data

$C_{11}H_{12}N_2S$
M_r = 204.29
Triclinic, $P\overline{1}$
a = 6.6159 (7) Å
b = 8.507 (1) Å
c = 9.468 (1) Å
α = 88.57 (6)°
β = 72.53 (9)°
γ = 80.89 (4)°
V = 501.7 (1) Å^3
Z = 2
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2941 reflections
θ = 1–25.1°
μ = 0.28 mm^{-1}
T = 298 K
Prism, colourless
0.45 × 0.30 × 0.15 mm

Data collection

KappaCCD diffractometer
φ scans
1581 measured reflections
1573 independent reflections
1476 reflections with $I > 3\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.2°
$h = -7 \rightarrow 0$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

R = 0.052
wR = 0.091
S = 1.00
1476 reflections
127 parameters
H-atom parameters constrained
$w = 1/[s^2(F_o^2) + 0.03F_o^2]$
$(\Delta/\sigma)_{max}$ = 0.028
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.7437 (7)	N10—C2	1.3635 (11)
S1—C14	1.8245 (8)	N10—C9	1.3801 (8)
N3—C2	1.3228 (10)	N10—C11	1.4640 (9)
N3—C4	1.3855 (10)		
C2—S1—C14	100.36 (4)	C2—N10—C11	127.57 (6)
C2—N3—C4	103.52 (7)	C9—N10—C11	126.32 (6)
C2—N10—C9	106.09 (6)		

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *maXus* (Mackay *et al.*, 1999); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *maXus*.

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