

2,3,4,5-Tetrahydro-*r*-2,*c*-4-diphenyl-1,5-benzothiazepine

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Key indicators

Single-crystal X-ray study

T = 150 K

Mean σ (C–C) = 0.004 Å

R factor = 0.054

wR factor = 0.131

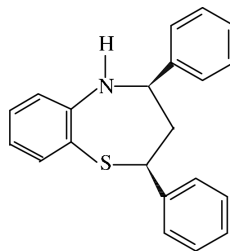
Data-to-parameter ratio = 18.1

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

The single-crystal X-ray diffraction study of the title molecule, C₂₁H₁₉NS, confirms the twist–boat conformation of the thiazepine ring. The two phenyl groups are equatorially oriented with respect to the thiazepine ring.

Comment

The molecular structure of the title compound, (I), resembles that of the calcium antagonistic drug, diltiazem (Kojic -Prodic *et al.*, 1984) with respect to the presence of a 1,5-benzothiazepine ring. A search of the Cambridge Structural Database (Allen & Kennard, 1993) for 2,4-substituted 1,5-benzothiazepine derivatives revealed that only two structures without amide or azomethine link had been reported. The crystal and molecular structure of (I) was determined to assign its molecular conformation and the orientations of the substituents on the tetrahydrothiazepine ring.



(I)

As confirmed by the X-ray analysis, the seven-membered ring in (I) adopts a twist–boat conformation, with the pairs of atoms N5/C11 and S1/C10 being oppositely oriented with respect to the C2/C3/C4 plane. The torsion angles around C2–C3 and C3–C4 are very close to the value of 45° reported for the corresponding torsion angle in the ideal twist–boat conformation of cycloheptane (Hendrickson, 1961). There are considerable torsional distortions in the other bonds of the seven-membered ring but the overall twist–boat conformation is maintained. Such distortions are characteristic of similar molecules (Kojic-Prodic *et al.*, 1984; Kumaradhas & Nirmala, 1997; Kumaradhas & Nirmala, 1996; Kumaradhas *et al.*, 1995). The *sp*² and *sp*³ hybridized states for C10 and C2, respectively, account for the difference in the S–C bond lengths in the title molecule (Table 1). The sum of the bond angles around N5 is found to be 346.2 (2)°, indicating some deviation from planarity of the bonds around nitrogen. The equatorial orientations of the two phenyl groups are deduced from the corresponding torsion angles, which are close to 180°. The diequatorial orientations of the two phenyl groups confirm their *cis* configurational dispositions. The N–H bond is not involved in any hydrogen-bonding interaction.

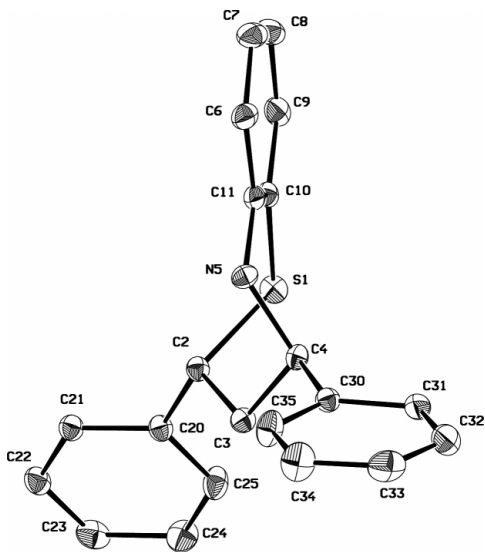


Figure 1

A view of the title molecule, showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels. H atoms have been omitted for clarity.

The intermolecular $S1 \cdots N5(x, -1 + y, z)$ distance of 3.490 (3) Å is greater than the sum of the van der Waals radii of the two atoms, indicating no strong non-bonding interactions between the two atoms.

Experimental

The reduction of the double bond in 2,3-dihydro-2,4-diphenyl-1,5-benzothiazepine (Stephens & Field, 1959) with sodium borohydride yielded the title compound, (I). Diffraction-quality crystals were obtained by recrystallizing the crude product from ethanol.

Crystal data

$C_{21}H_{19}NS$	$D_x = 1.231 \text{ Mg m}^{-3}$
$M_r = 317.43$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1726 reflections
$a = 13.4782 (15) \text{ \AA}$	$\theta = 10\text{--}28^\circ$
$b = 6.0737 (7) \text{ \AA}$	$\mu = 0.19 \text{ mm}^{-1}$
$c = 21.918 (3) \text{ \AA}$	$T = 150 (2) \text{ K}$
$\beta = 107.327 (3)^\circ$	Needle, colorless
$V = 1712.9 (3) \text{ \AA}^3$	$0.44 \times 0.09 \times 0.08 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART 1K CCD diffractometer	4168 independent reflections
ω - 2θ scans	1824 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.087$
$T_{\text{min}} = 0.922$, $T_{\text{max}} = 0.985$	$\theta_{\text{max}} = 28.3^\circ$
10 671 measured reflections	$h = -17 \rightarrow 17$
	$k = -8 \rightarrow 7$
	$l = -29 \rightarrow 23$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2]$
$wR(F^2) = 0.131$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.86$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4168 reflections	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
230 parameters	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1—C10	1.770 (3)	C4—N5	1.476 (3)
S1—C2	1.842 (2)	C4—C30	1.509 (3)
C2—C20	1.515 (3)	N5—C11	1.395 (3)
C2—C3	1.524 (3)	C10—C11	1.402 (3)
C3—C4	1.541 (3)		
C10—S1—C2	102.58 (12)	C11—N5—C4	121.5 (2)
C20—C2—C3	114.2 (2)	C9—C10—C11	119.0 (2)
C20—C2—S1	106.58 (16)	C9—C10—S1	115.8 (2)
C3—C2—S1	112.07 (17)	C11—C10—S1	125.2 (2)
C2—C3—C4	111.7 (2)	N5—C11—C6	117.8 (2)
N5—C4—C30	109.0 (2)	N5—C11—C10	123.8 (2)
N5—C4—C3	109.64 (19)	C6—C11—C10	118.3 (2)
C30—C4—C3	114.2 (2)		
C10—S1—C2—C3	151.07 (17)	C30—C4—N5—C11	138.0 (2)
C10—S1—C2—C3	−83.4 (2)	C3—C4—N5—C11	−96.3 (3)
C20—C2—C3—C4	167.2 (2)	C2—S1—C10—C11	32.2 (2)
S1—C2—C3—C4	45.9 (3)	C4—N5—C11—C10	38.4 (3)
C2—C3—C4—N5	45.3 (3)	S1—C10—C11—N5	0.7 (3)
C2—C3—C4—C30	168.0 (2)		

The position of the H atom attached to N5 was located from a difference map and subsequently refined along with its displacement parameter. All the other H atoms were included in their appropriately calculated positions. While the C—H distances were constrained by a riding model, the displacement parameters of these H atoms were tied to a free variable.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ZORTEP (Zsolnai, 1997); software used to prepare material for publication: SHELXL97 (Sheldrick, 1997) and PARST96 (Nardelli, 1983).

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